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COMMERCIAL
LABORATORY NOTES

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COMMERCIAL LABORATORY NOTES

Vol. I.

By

C. L. PETTY

PUBLICATION OF THE

Dayton Metal Products Company

RESEARCH DIVISION

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Preface

The methods given in this book have been very carefully selected from various sources and only those that have proven satisfactory are included. With a few exceptions these notes have been used by the author, repeatedly, and the results obtained are good. They are stated simply and in the form that will be of the most assistance to the analytical chemist.

It has been the experience of the author that when one is called on to do work along lines that are somewhat different from his regular work, he does not have the inclination or the time to look through the literature to find a method that will suit his particular need; and even though he did take the time, he is often confused with the mass of material that is presented to him and, furthermore, is at a loss relative to the selection of the method most applicable.

This volume covers the following subjects sufficiently well to be of great value to analytical chemists: Non-Ferrous Alloys, Paint, Oil, Rubber, Steel and Iron, as well as a collection of miscellaneous materials. These methods as a whole are the ones in daily use by the Pennsylvania Railroad Co. at Altoona, Penn., with some slight additions, subtractions, and modifications, to make the methods more comprehensive; consequently due credit must be given the Pennsylvania Railroad laboratory as the originator of many of the methods included in these notes. These methods have also stood the test of the World War in the laboratories of the Dayton Metal Products Co., Research Division, Dayton, Ohio.

In almost every laboratory the conditions are different. One has to modify methods to a certain extent in order to be able to use the available apparatus, and to meet other conditions that may arise. Also the laboratory in question may have an additional test to make on a certain analysis, that is of primary interest to him, that is not described herein. In this case this book supplies a means whereby the chemist may insert his methods and always have them at hand when needed. Notes that are scattered or written down in a half dozen books have very little value, as too much time is wasted in looking for them. It is the carefully recorded and filed notes that are of the most value.

This book is put out in the loose leaf form for several reasons: It permits the chemist to insert any method found to be good by experience, and any other method that he may have occasion to use. It may always be kept up to date by extracting methods that have become obsolete and replacing them with the new ones. Books that are bound do not present this advantage; the only course left open is to buy a new edition when the methods are superseded, by better ones, which they do very rapidly in these times of rapid scientific advancement. In other words, this book forms a sort of nucleus around which the individual chemist may build for himself a collection of notes that will be invaluable to him, and one that will always be up to date. The initial cost of this volume is very small compared with the cost of maintaining a library up to date that will give the workable information that is given herein.

The author wishes to thank those who have assisted in the compilation of this volume. Special mention of Mr. H. C. Mougey is necessary, for his criticism and assistance in proofreading.

C. L. PETTY.

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COMMERCIAL LABORATORY NOTES

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Method of Analysis of Aluminum and Aluminum Castings

SILICON

Silicon is determined in the same manner as in the determination of silicon in steel.

MANGANESE

Dissolve a 1 gram sample in 2 c. c. sulphuric acid, 75 c. c. water and 35 c. c. concentrated nitric acid, C. P., and proceed as in the determination of manganese in steel by the sodium bismuthate method

IRON

Dissolve a 1 or 2 gram sample in 5 grams potassium hyrdoxide and 35 c. c. water. Filter, wash with hot water, dissolve the residue which contains the iron in 10 c. c. sulphuric acid and 30 c. c. water. Run through a Jones' reductor and titrate with a standard solution of potassium permanganate.

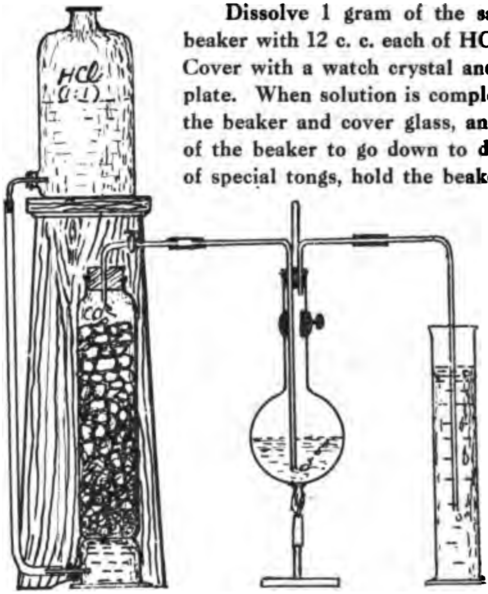
COPPER AND ZINC

Dissolve 5 gram in 5 grams potassium hydroxide and 20 c. c. water. Boil until in solution. Cool, dilute, filter and wash with hot water. Dissolve the residue in 12 c. c. nitric acid, (Sp. Gr. 1.42,) dilute to 225 c. c. and electrolyze for copper.

Add 1 c. c. sulphuric acid (Sp. Gr. 1.84 to electrolyte, evaporate to fumes of sulphuric acid. Add filtrate from above, make alkaline with potassium hydroxide and boil off any ammonia which may be present. Add 4 grams of Rochelle salts, boil, cool, dilute to 275 c. c. and electrolyze for zinc on a copper coated electrode.

Other impurities which may be present must be treated individually according to composition.

Determination of Tin in Bronzes



Dissolve 1 gram of the sample in a 250 c. beaker with 12 c. c. each of HCl, HNO$_3$ and H$_2$SO Cover with a watch crystal and place on the stea plate. When solution is complete remove and rin the beaker and cover glass, and allow the conten of the beaker to go down to dryness. With a pa of special tongs, hold the beaker over a free flam keeping it in co stant motion t prevent spatteri until dense fume are being drive off. Cool and add 5 c. c. of HCl, (1.08 Sp. Gr.) cover an stand the beaker o the steam plate u til all of the res due is in solutio except lead chlo ide. Remove th beaker, allow it contents to cool u til the beaker can be held in the hand. Add 2 to 3 grams of low ca bon content steel to precipitate all copper. When the solution is com pletely discolored, rinse down the sides of the beaker and cover glas and allow the beaker to stand 2 or 3 minutes longer. Filter throug a rapid paper into a round bottom 500 c. c. flask. Wash the contents o the beaker twice with 50 c. c. HCl (1.088 Sp. Gr.) and 150 c. c. H$_2$ Add 1 gram pulverized C. P. Antimony and bring to a boil. Place th flask and contents in boiling water for ten minutes, shaking occasion ally; then connect up the flask to a CO$_2$ apparatus as shown in th sketch, and start the passage of the gas through the liquid; bring t boil with burner and continue to heat at incipient ebullition for fiv minutes, passing gas throughout the treatment.

Remove the burner, and cool down in ice until thoroughly col Continue the passing of the gas. (7 to 10 minutes.) Disconnect, ad 2 c. c. freshly prepared starch solution, rinse tubes and walls of flas and quickly titrate with approximate N/$_{10}$ iodine solution (accuratel standardized) to blue color.

Factor × number of c. c. × 100 = per cent. Sn.

Analysis of Pig Lead

Dissolve a 1 gram sample in 10 c. c. nitric acid and 15 c. c. water, in a liter beaker.. When in solution remove the watch glass cover and evaporate to dryness on the steam plate. Dissolve in 50 c. c. water*. If the solution is clear and there is no residue, add 10 c. c. of ammonia and 25 c. c. of 50 per cent acetic acid. Dilute to about 700 c. c., boil, add 25 c. c. of potassium bichromate solution (36 grams potassium bichromate dissolved in 1 liter water) and boil a few minutes, allow the precipitate to settle on the steam plate, and filter onto a good asbestos pad in a Gooch crucible. In general, the number of c. c. of $K_2Cr_2O_7$ solution required in one-fourth the number of centrigrams of lead present. The Gooch crucible and pad are previously dried, ignited and weighed. Dry the crucible and contents on a platinum lid over a low flame. Gradually increase the heat until the precipitate is dry and becomes a cherry red while hot. Cool in the desiccator and weigh. The increase in weight is lead chromate.

Lead chromate × .641 gives amount of lead (metallic).

*If there is any residue when lead nitrate is dissolved in water, it must be filtered off and determined.

In ordinary commercial practice, Pig Lead is bought for certain purposes and usually under specifications and the manufacturer is not materially concerned with impurities unless they affect his product in some way. He demands a certain grade of lead and checks it up to see if he is getting what he paid for. Consequently, the individual chemist must be guided by his particular need in his search for impurities. Of course, when an unknown or miscellaneous sample is received, it must be analyzed completely.

Impurities: Copper, Tin, Antimony, Bismuth, (Gold and Silver).

Copper, Antimony and Tin—Run as in the analysis of Lining Metal, Method No. 9.

Gold and Silver—Make an assay on one assay ton (29.166 grams) and the weight of the resulting bead in milligrams equal ounces (troy) per ton of metal.

Bismuth—Determine as in the analysis of Bismuth Method No. 17.

Lead and Copper in Brasses and Bronzes

Weigh 1 gram into a 250 c. c. beaker, add 7 c. c. HNO_3 and 4 c. c. H_2O, cover quickly. Set the beaker on the hot plate under the hood. When all violent action is over, remove the cover glass, rinse down walls of beaker with water and allow the contents of the beaker to go down to dryness, but do not allow to bake, as there is danger of the formation of difficulty soluble oxides. Take up the residue in 50 c. c. of (1:5) HNO_3. Set the beaker on the hot plate until the solution of the lead and copper salts is complete. Remove, and filter through a 9 c. m. tight filter paper, catching the filtrate in a 250 c. c. beaker; wash with hot water until a few drops of the filtrate evaporated on a platinum foil leaves no residue.

Cover the filtrate in a 250 c. c. beaker, with a watch crystal and set aside for the present. Place the filter paper and the metastannic acid back into the original beaker. Add 10 c. c. water and 1 gram of NaOH. Heat on the steam plate for five to ten minutes, then add 100 c. c. of the following solution:

Ammonium Oxalate	10 grams
Oxalic Acid	200 grams
Water (distilled) to make	2000 c. c.

Boil for a few minutes, when all tin should be in solution. Now pass H_2S gas through this hot solution for ½ hour to precipitate all traces of lead and copper in the metastannic acid. The solution containing the tin is filtered into a 250 c. c. beaker, catching the sulphides of lead and copper on a 7 c. m. filter paper. Wash thoroughly with distilled water, through which H_2S gas has been passed. Transfer the precipitate and paper to a porcelain crucible and ignite gently. Burn off the paper in the open crucible. When the paper has been burned, add 5 drops of HNO_3 conc. to the residue in the crucible, warm to dissolve traces, and wash all the contents of the crucible into the beaker containing the copper and lead. Dilute up to 200 c. c. and electrolyze over night for copper and lead.

According to our practice, good results are obtained as follows:

Use of stationary electrodes sheets of platinum foil made into cylinders of approximately 1" in diameter and 2" long for the cathode and 1⅞" in diameter and 2" long for the anode. These electrodes are fitted with platinum stems about 5" long and projecting 3" above the electrode. The large size electrode is used for the anode in order to provide sufficient surface for PbO_2 to deposit, especially in bronzes, where the lead may at times be as high as 30%. The electrolysis is carried on by completely covering the electrodes with the solution, and electrolyzing over night with a current of about .5 ampere. To test for complete precipitation, dilute with enough water to raise the volume about 1/16" and note whether the freshly exposed surface of the stem remains bright. A more delicate test is to pipette out some of the clear liquid and test it with ammonium hydroxide or potassium ferrocyanide. It is advisable to add a few drops of sulfuric acid in the morning and electrolyze for about one hour with 1 ampere to remove the last traces of Copper.

The electrodes are washed with two beakers of water in the following manner:

With the left hand hold the beaker containing the electrolyte, and with the right hand remove the support under the beaker containing the electrolyte. Then get a beaker of wash water in the right hand and quickly remove the beaker containing the electrolyte and replace it with the one in the right. In a similar manner wash with a second beaker of wash water. The electrodes may then be disconnected and washed in alcohol and dried in an oven at about 100° C. for about 10 minutes and weighed. In case the electrode contains over .2 gram of lead oxide, ti is desirable to dry it at 180° C. for about 2 hours. This must be done on a weighed watch glass to prevent mechanical loss of lead oxide.

$PbO_2 \times .866 = Pb.$

The PbO_2 may be cleaned from the electrode by dipping it in a jar containing 1:1 HNO_3 containing oxalic acid. The copper may be stripped in straight 1:1 HNO_3.

NOTE: Be sure to cover the beaker during the electrolysis, as the poles to which the platinum electrodes are attached may be copper, and when the electrolysis is going on rapidly, nitric acid and water is thrown mechanically upon these poles, so in time they become corroded. Consequently there is danger of copper salts dropping into the beaker, causing high results.

2. Instead of filtering off the metastannic acid, the solution, when taken up in (1:5) nitric acid, may be placed on the electrolytic machine and electrolyzed for lead and copper. If the results are low, it will be necessary to run for traces. In this case, filter off the metastannic acid and proceed as prescribed in the foregoing.

It will also be necessary to filter off the mestannic acid if a zinc determination is desired.

IMPORTANT. Save all the electrolytes until the sample has been reported, because it may be necessary to determine traces of lead, copper, antimony, zinc, nickel, etc.

In the presence of several per cent or more of tin, the SnO_2 left by dissolving the sample in nitric acid is contaminated with lead and copper and other impurities. Consequently, for accurate work, the above method is desirable. If the tin is low, or if very accurate results are not required, the tin can be determined by filtering off the SnO_2 left after dissolving the sample in nitric acid.

Method of Determining Zinc and Nickel in Brass and Bronze

Transfer the electrolyte from the copper and lead determination, (Method 4) into a large porcelain evaporating dish, add 4 c. c. sulphuric acid C. P., 1.84 Sp. Gr., and evaporate to fumes of sulphuric acid. Dilute with distilled water to about 300 c. c. volume, make strongly alkaline with potassium hydroxide and boil until all the ammonia fumes are driven off, diluting and adding more potassium hydroxide, if necessary. Cool and place on a steam plate in a hood, and add at once about 15 c. c. strong bromine water or a few drops of bromine. Stir until the precipitation of nickelic hydroxide takes place, and boil or allow to stand on the steam plate until the bromine vapors are driven off. Dilute with distilled water to about 300 c. c. volume and filter, washing with hot water, traces of copper remaining from the copper determination will be precipitated with nickel. Boil the filtrate until the last traces of bromine are driven off; cool, add about 4 grams Rochelle salts, heat to complete solution, cool and electrolyze for zinc on a weighed copper coated electrode. Remove from the electrolyte apparatus, wash, dry and weigh as usual.

If nickel is not present the zinc may be determined without the treatment with bromine.

NICKEL ELECTROLYTIC METHOD

Dissolve the nickelic hydroxide and traces of copper from the filter with 10 c. c. hydrochloric acid, C. P., 1.20 Sp. Gr., and 10 c. c. water, into the evaporating dish, wash with hot water and make a volume of about 150 c. c. Boil to drive off any bromine present and evaporate to about 100 c. c. volume. Wash into a 180 c. c. beaker, make alkaline with ammonia, add 50 c. c. of ammonia, C. P., .90 Sp. Gr., in excess and electrolyze at about 70 degrees Fahrenheit. Remove from the electrolytic apparatus, wash, dry and weigh as usual.

The copper which may be present is deposited with the nickel. Dissolve the deposit on the electrode in 3 c. c. nitric acid, C. P., 1.42 Sp. Gr., dilute to about 100 c. c. and electrolyze for copper. Subtract the weight of the copper deposit from the weight of the combined deposit of copper and nickel and report the difference as nickel

NICKEL DIMETHYLGLOXIME METHOD

In case nickel alone is desired, it can be determined by the following method, without removing the zinc, or the dimethylgloxime method may be used instead of the electrolytic method, after dissolving the nickelic hydroxide obtained above, in hydrochloric acid.

The electrolyte, after removing lead and copper by electrolysis, is made slightly ammoniacial, then neutral and slightly acid with HNO_3 The neutral or slightly acid solution is diluted so that not more than .1 gram Co or Ni is present in 100 c. c. of the solution, heating nearly to boiling and add an excess of alcoholic solution of dimethylgloxime (20 c. c. of a 10% solution.) Ammonia is then added cautiously until the solution smells slightly of ammonia. While still hot, the solution is filtered through a Gooch crucible, washed with hot water and dried at 110 to 120 degrees Centigrade for 45 minutes.

Factor=.20316×100×wt. of precipitate=% of Ni.

Co, Zn and Mn do not interfere. In the presence of Fe, tartaric acid should be added to prevent its being precipitated by ammonia.

Reference: Treadwell-Hall, Vol. II, Page 125.

Analysis of Ingot Copper

COPPER

Weigh a 1 gram sample into a 200 c. c. beaker and dissolve in a mixture of 10 c. c. of water, 5 c. c. sulphuric acid, and 3 c. c. of nitric acid. Evaporate to a syrupy consistency, dilute to 180 c. c., heat nearly to boiling and place on the revolving electrode apparatus and electrolize with a current of 4 amperes for one hour when all the copper should be deposited if all the conditions are followed.

A gauze cathode is used and a spiral anode. The gauze allows freer circulation of the solution and provides a larger surface on which the copper may deposit.

For more accurate results, a larger sample should be taken and the copper should be plated out on a large stationary electrode, using a current of approximately .3 ampere.

ARSENIC

Dissolve 10 grams of the sample in 40 c. c. nitric acid (Sp. Gr. 1.42). Boil to expel red fumes and dilute to 300 c. c. Neutralize with ammonia until there is just enough copper hydrate formed to cover sparsely the bottom of the beaker. Add about 1 gram of ferric ammonium sulphate in aqueous solution. Boil one-half hour. Filter and wash with hot water. The iron precipitate containing the arsenic is dissolved in warm hydrochloric acid and transferred to the distillation flask described in the Determination of Arsenic in Pig Tin, Method No. 12. The remainder of the determination is exactly in accord with Method No. 12.

LEAD

Dissolve a 5-gram sample in a mixture of 20 c. c. of water and 20 c. c. of nitric acid. Evaporate nearly to dryness. Dilute up to 200 c. c. and electrolyze for lead with a current of about ¾ ampere, disregarding the copper altogether. Electrolysis will be complete in about 3 hours.

NOTE: In ordinary commercial practice, copper is bought for certain purposes and usually under specificatons. For electrical use it is frequently desirable to determine the resistance of the copper, since exceedingly small amounts of impurities affect the resistance greatly. The purchaser requires a certain grade of copper for his use and checks it up to see if he is getting what he paid for. Consequently, the individual chemist must be guided by his particular need in his search for impurities. Of course, when an unknown or miscellaneous sample is received it must be analyzed completely. For the complete analysis of impurities in copper, see Technical Analysis of Brass by Price and Meade.

Method of Determining Phosphorus in Phosphor Bronze

APPARATUS AND REAGENTS

The apparatus required by this method needs no special comment.

The aqua regia is made by equal parts nitric and hydrochloric acid by volume, both concentrated C. P.

The C. P. ammonia is obtained in the market, Sp. Gr. 0.90.

The ammonium sulphide solution is made by treating C. P. ammonia, Sp. Gr. 0.90, with hydrogen sulphide until no further absorption takes place, and then adding two-thirds as much by volume of the same ammonia solution. It is only slightly yellow in color, and may usually be obtained in the market.

The ammonium sulphide wash water is made by adding three parts of distilled water to one part of the above solution, both by volume.

The magnesia mixture is made by dissolving 66 grams of crystallized C. P. magnesia chloride and 168 grams of C. P. ammonium chloride in 780 c. c. of distilled water, and adding 420 c. c. of C. P. ammonia, Sp. Gr. .900. Allow to stand two days and filter.

The ammonia wash water is made by adding to 800 c. c. of distilled water 200 c. c. of C. P. ammonia, Sp. Gr. 0.90, and 25 grams of crystallized C. P. ammonium nitrate. Filter before using.

The dilute hydrochloric acid is made by adding 1 part concentrated C. P. acid, Sp. Gr. 1.20, to 4 parts distilled water, both by volume.

OPERATION

Put 1 gram of fine borings in a beaker about 2½ inches in diameter by 3½ inches high, and add 25 c. c. of aqua regia. Cover the beaker and allow to dissolve; then heat to near boiling point of the solution for half an hour. Add 25 c. c. of distilled water and then 20 c. c. of concentrated C. P. ammonia, Sp. Gr. 0.90. Then add 50 c. c. of ammonium sulphide. The principal portion of the copper and lead is precipitated as sulphides, and the tin and phosphoric acid are in solution. which is a clear yellow. Digest at a temperature near the boiling point of the solution for 20 minutes. Allow to settle, then filter through a 9 c. m. filter into an Erlenmeyer flask, holding about 300 c. c. The filtration and washing is best managed as follows: Pour the clear liquid on the filter, and allow most of it to run through. Then pour the remaining liquid with the precipitate on the filter, and allow it to drain thoroughly. Put the filter with the precipitate on it back into the beaker, and add 50 c. c. of the ammonium sulphide wash water. Warm and stir occasionally for 10 minutes to secure as complete solution and diffusion of the soluble material as possible; then pour everything on another filter and allow it to drain thoroughly. Then wash on filter with about 50 c. c. more of the ammonium sulphide wash water. This gives a volume of filtrate of about 200 c. c. Add now to this filtrate 10 c. c. of magnesia mixture, and shake to secure uniform diffusion of the precipitate. Put the flask in ice water and allow to stand in same with occasional agitation for two hours. Filter through a 7 c. m. filter and wash with ammonia water only until the washings react slightly with silver nitrate Then add 10 c. c. of dilute hydrochloric acid to the flask and so manipulate that the liquid touches all parts of the inside of the flask and dissolves any adhering precipitates. Then with the liquid in the flask dissolve the precipitate on the filter, allowing the solution to run into a smaller beaker. Wash the flask and filter with the dilute hydrochloric acid until the volume of the filtrate is about 30 c. c. Add now 5 c. c. of the magnesia mixture and 10 c. c. of concentrated C. P. ammonia, 0.90 Sp. Gr.; agitate by stirring, put in ice water, and allow to stand with occasional stirring for two hours. Filter on a 5 c. m. filter, wash with ammonia wash water, until the washings react only slightly opalescent with silver nitrate. Smoke off filter, or filter on a Gooch crucible if the operator so desires and ignite and weigh. Ignite until the precipitate is white, and weigh.

CALCULATIONS

The atomic weights used are magnesium, 24.32; phosphorus, 31; oxygen, 16. The molecular formula of magnesia, pyrophosphate used is $Mg_2P_2O_7$.

Since 27.86 per cent. of the magnesium pyrophosphate is phosphorous, the amount of phosphorus in the precipitate may be obtained by the proportion a:b::0.2793:x. in which "A" represents the amount of phosphor bronze taken to start with, expressed in grams; B, the magnesium pyrophosphate obtained in grams; and x the phosphorus sought, which will likewise be in grams. Then since the above proportion gives the actual amount of phosphorus in 1 gram of phosphor bronze, it is obvious that the per cent. of phosphorus, that is, the amount in 100 grams or parts, will be 100 times this amount. Where 1 gram is taken to start with the following brief rule may be used: Express the weight of magnesium pyrophosphate found in grams, move the decimal point two places to the right, and multiply by the decimal 0.2786. Thus if the magnesium pyrophosphate found is 0.0304 gram, the per cent. of phosphorus is (3.04 x 0.2786) 0.837 per cent.

NOTES AND PRECAUTIONS

It will be noted that this method oxidizes the phosphorus by means of nitric acid, with hydrochloric acid present to hold up the tin: separates the phosphoric acid from the lead and copper by means of ammonium sulphide, and precipitates the phosphoric acid as magnesium ammonium phosphate in presence of tin and ammonium sulphides, the precipitate being purified by a second precipitation before weighing.

The aqua regia used contains more nitric acid than is customary for this reagent. Nitric acid alone would possibly be as good or perhaps better to oxidize the phosphorus, but experience shows that the metastannic acid formed when nitric acid alone is used dissolves in the ammonium sulphide with some difficulty. Incomplete solution would, of course, result in loss of phosphorus. Accordingly some hydrochloric acid is used to bring the tin into solution. With the method as recommended, there is no difficulty with the tin.

Heating the aqua regia solution for half an hour after the metal is in solution secures complete oxidation of the phosphorus, and the addition of water dilutes the acid sufficiently so that the strong ammonia water can be added without too violent reaction.

If the ammonium sulphide used is made as recommended, very little if any of the copper sulphide is dissolved. Strong yellow sulphide of ammonium gives more difficulty from this cause; and if the yellow sulphide is used, the first ammonium magnesium phosphate precipitate may be contaminated with copper sulphide, which has separated during the two hours in the ice water. This copper sulphide may not cause subsequent difficulty, but it is better not to have it present.

The 20 minutes digestion after the ammonium sulphide is added and the filtration and washing recommended, successfully removes the phosphorus from the lead and copper sulphides. An examination of these sulphides by decomposing them with nitric acid, separation of the lead sulphide, then separation of the copper as sulphide in acid solution by means of hydrogen sulphide after neutralization of the free acid with ammonia, concentration of the filtrate to small bulk, and testing with molybdate of ammonia solution shows only a trace of yellow precipitate.

It is desirable to have the bulk of solution in which to precipitate the phosphoric acid by magnesium mixture as small as may be on acccount of the possible solubility of this precipitate. At the same time the lead and copper sulphides are so gelatinous that complete washing on the filter is difficult. The proceedure recommended apparently secures the result desired with the least amount of wash water.

The following experiment has been made on this point. When the ammonium sulphide solution is ready to filter, the total bulk due to reagents added should be 120 c. c.; but on account of evaporation and decomposition the actual bulk was 109 c. c. Of this 101 c. c. ran through the filter after the clear liquid and precipitate had been put on the filter as directed. After putting the precipitate and filter back into the beaker, adding 50 c. c. ammonium sulphide wash water, and digesting covered for 10 minutes, the bulk of solution, including the filter, was 59 c. c. Of this 48 c. c. ran through the second filter. Neglecting the volume of the precipitate, and assuming that the phosphoric acid is uniformly disseminated in the liquid, it is evident that eight one-hundred and ninths of the phosphorus is left behind after the first filtration and eleven fifty-ninths of this after the second filtration. Reducing these fractions, it appears that after all that will has run through the second filter, only 1.37 per cent. of the phosphorus is left behind. As phosphor-bronze usually contains less than 1.00 per cent. of phosphorus, it is obvious that only about one hundredths of a per cent. of phosphorus remains to be washed out. The 50 c. c. of wash water recommended is apparently abundant for this purpose. In the above experiment the measurements, when the precipitate was present, were made in the beaker, and must be regarded as close approximations only.

The use of a flask for the first precipitation offers some advantages over a beaker, the principal one being that the ammonium sulphide is less exposed to the air and consequently undergoes less change, with resulting less probability of throwing down free sulphur or traces of sulphide of copper than if a beaker is used. The flask should be covered with a small watch glass, but it is not necessary to use a cork or glass rod for stirring.

Over-washing of the ammonium magnesium phosphate is to be avoided as carefully as under-washing. The directions given should be closely followed.

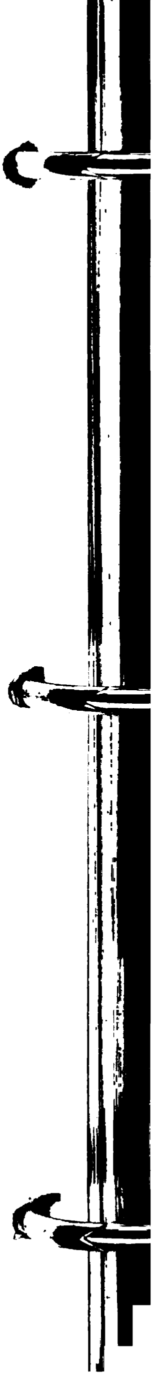
The precipitation of the phosphoric acid by excess of magnesia mixture, the presence of the tin and ammonium sulphides, seem to be as satisfactory as in the presence of chloride of ammonium alone. An examination of the filtrate and washings from the first precipitation on evaporation nearly to dryness, taking up with nitric acid and just enough hydrochloric acid to hold up the tin, and testing with molybdate solution, shows only a trace of yellow precipitate.

It is not advisable to weigh without the second precipitation. Although careful manipulation and the use of colorless ammonium sulphide may avoid contamination from copper sulphide, there is always danger of free sulphur in the ammonium magnesium phosphate. For good work the second precipitation should never be omitted.

Ammonium magnesium phosphate is liable to be reduced during the ignition of the filter and thus lead to slightly low results. To obviate this difficulty, the filter and precipitate are put into the crucible wet, and the filter "smoked off" and then burned. The "smoking off" consists in applying the heat to the wet material in the crucible so slowly that the volatile matter of the filter passes off without ignition, free access of air being maintained at the same time. To accomplish this, fold up the wet filter with the precipitate in it, and place it in the crucible. Put the crucible on the triangle as in ordinary ignitions, and leave the cover off. Then heat the open end of the crucible slowly. The filter and precipitate gradually dry, and soon the parts of the filter in contact with the crucible begin to distil off the volatile matter at low heat, even before the whole is dry. This process goes on if the flame is properly adjusted, until in a little while everything that is volatile at a low temperature has passed away, and the precipitate, with a black envelope of carbonaceous matter is left. When this is the case the temperature can be raised, the lamp moved back to heat the bottom of the crucible, and the carbon burned off completely. Usually when the temperature is raised, the black envelope of carbonaceous matter falls away from the precipitate and is readily consumed. By this method of ignition the material is a little longer time in the crucible than the old method of previously dried precipitate, but the danger of reducing the precipitate is believed to be very much diminished. The small amount of nitrate of ammonia wash water left in the filter paper facilitates this operation.

When a bronze contains only small amounts of phosphorus, it is advisable to start with 2 to 5 grams. The manipulation and proportions of reagent are, however, the same except that 75 c. c. of ammonium sulphide should be used for the first addition, and 75 c. c. of of ammonium sulphide wash water for the second addition, and about 100 c. c. of the same wash water for washing on the filter. This gives a bulk of about 300 c. c. for the first precipitation.

It seems probable that the method described above in careful hands will give results accurate to about one hundredth of a per cent. although it is not rare that duplicate determinations on the same sample differ two hundredths. Where proper care is given to each point it takes about seven hours to get a result.



Determination of Zinc in Bronzes

The electrolyte and washings from lead and copper determinations is transferred to a large evaporating dish, add 4 c. c. sulphuric acid and evaporate to fumes of sulphuric acid. Place the dish aside to cool, add 100 c. c. of water + 8 grams of NaOH. Boil until all ammonium salts are expelled. Transfer the solution to a 250 c. c. beaker, making sure to wash the dish well. Add 4 to 5 grams of Rochelle salts, boil and place on electrolytic apparatus and electrolyze on a weighed copper coated electrode, with a strong current. All zinc will be completely electrolyzed in 4 hours.

If it is desired to obtain the results in a short time, the hot solution may be electrolyzed on the revolving electrode machine, with a strong current. In this case all zinc will be down in from twenty to thirty-five minutes.

Method of Analysis of Lining Metal and Babbitt

COPPER AND LEAD

To 1 gram of the alloy in a 250 c. c. Erlemeyer flask, add 13 c. c. of a tartaric acid solution ($333^1/_3$ grams of C. P. tartaric acid, to 1 liter distilled water. For babbit or other alloys high in tin, use more tartaric acid solution. In general the number c. c. of tartaric acid solution is 30 to 40 times the grams of tin. Heat to boiling and then add 3 c. c. of nitric acid, C. P., (1.42 Sp. Gr.) drop by drop. Heat on the steam plate until solution is complete. Then add 4 c. c. concentrated sulphuric acid, boil gently and at the same time blow off nitric acid fumes with a bent glass tube, being careful not to char the tartaric acid. When operation is completed, remove quickly and add about 50 to 60 c. c. water. Filter and wash with cold water by decantation, into a No. 4 beaker. This beaker contains copper and traces of lead, which should be set aside for the present.

Take the filter paper containing lead sulphate and return it to the Erlemeyer flask, add 10 c. c. ammonium hydroxide and 25 c. c. acetic acid, (50 per cent.) and warm on the steam plate until solution is complete. Then filter into a 700 c. c. beaker and wash well with hot water. Transfer these filter papers to the number 4 beaker containing copper and trace of lead, so as to catch any lead that might be left in them.

The filtrate in the 700 c. c. beaker is diluted to two-thirds capacity of beaker, brought to boil and 25 c. c. of potassium bichromate are added (36 grams potassium bichromate to 1 liter water.) Boil for two minutes longer and place in a warm place over night. Filter the precipitate on weighed Gooch crucible, wash well with hot water by decantation and dry to constant weight and calculate lead. To the number 4 beaker add enough caustic soda to just make alkaline, bring to boil and add 20 c. c. sodium sulphide (1 ℔ crystals to 1 liter water.) Boil for a few minutes, then set the beaker on the steam plate until the sulphides settle.

Now take the number 4 beaker containing sulphides of copper and lead, filter and wash well. Return precipitate and filter papers to beaker, add a mixture of 10 c. c. nitric acid and 25 c. c. water and place on steam plate until sulphides are in solution, or in other words until papers are white. Then filter into a 250 c. c. beaker, wash well and electrolyze for the copper and lead.

ANTIMONY

Weigh one gram of the alloy in a 250 c. c. Erlemeyer flask, (Jena) add 10 c. c. concentrated sulphuric acid and about 5 grams of potassium sulphate. Dissolve over the bare flame, holding with crucible tongs and at the same time agitating the contents to prevent any possible bumping or loss, mechanically. The contents will at first be black and then whiten. Keep agitating over flame until contents are white or very light gray, the atmosphere in the flask is quite clear, and dense white fumes of sulphuric anhydride come off freely. Let it cool, then add 50 c. c. of a solution of tartaric acid containing 5 grams of tartaric acid, and then 10 c. c. of hydrochloric acid, concentrated; boil for about three minutes. Cool down under water tap and add 130 c. c. water and 10 c. c. concentrated hydrochloric acid. Cool down in ice water and titrate with a solution of potassium permanganate made up with two grams of the salt to one liter of water, and standardized against pure antimony.

TIN

Take this solution, after titrating for antimony, wash into a long neck 500 c. c. round bottom flask (Jena) with 50 c. c. concentrated hydrochloric acid, add about one gram of powdered antimony, (flour fine,) put into a bath of boiling water for about 15 minutes. Then connect up with a carbon dioxide gas supply, let gas flow through gently, having the end of the out-flow tube immersed in water to prevent any air drawing back and causing oxidation. Heat to boiling for five minutes, then cool down rapidly in ice and water, all the time having the carbon dioxide passing through the solution.

When cold titrate quickly with $N/_{10}$ solution of iodine. Standardize against pure tin. Use starch as an indicator.

For description of apparatus and further details concerning the determination of antimony and tin, see Low's Method in Journal of American Chemical Society for January, 1907. We found it necessary to make slight modifications to suit our own conditions.

In similar alloys containing appreciable quantities of copper, we find according to our experience that the copper may seriously affect the results obtained in the determination of tin. This difficulty can be overcome by precipitating the copper from a sulphuric acid and hydrochloric acid solution by means of metallic iron.

After filtering off the solution of tin and observing the conditions of acidity and bulk of solution as given above, reduction with antimony and subsequent titration with iodine solution, may be followed as before described.

In this case it will be necessary to make an independent determination of antimony. The influence of copper in this antimony determination needs more study. Several per cent. does not seem to affect the results seriously.

Method of Analysis of Battery Zincs

Determine copper, lead and iron in the same manner as in spelter.

MERCURY

Make up a mixture of 50 c. c. hydrochloric acid (specific gravity 1.20) and 150 c. c. water in a 300 c. c. beaker and allow to cool. To this add 5 grams finely ground or bored battery zinc. Allow to stand in a cool place, stirring occasionally. If all is not dissolved before night, add 25 c. c. of water and let stand over night. Filter on a 9 c. m. filter, wash thoroughly, squeezing the water and acid out of the spongy mercury black until it shows no indication of hydrochloric acid with silver nitrate.

Wash the mercury back into the beaker with a little water and add 5 c. c. of nitric acid (Sp. Gr. 1.42). Evaporate off excess of acid and water by placing on a sheet of asbestos placed on the gauze on the steam plate. Do not allow it to become too hot or to go to dryness. It is safe to remove when there is still about 1 c. c. of acid left and no sign of dryness is indicated.

Dilute up to 100 c. c. with cold water, add 2 c. c. hydrochloric acid (Sp. Gr. 1.20) and 25 c. c. phosphoric acid (Sp. Gr. 1.12). Stir it up and let it stand at room temperature over night in a dark place free from dust. Filter mercurous chloride onto a weighed asbestos pad in a 15 g. platinum Gooch crucible, washing by decantation several times and then wash the entire precipitate on the pad. Wash thoroughly until there is no reaction for hydrochloric acid in the filtrate. Allow the the suction to continue for 8 to 10 minutes until the mercurous chloride is air dried.

Place the crucible and contents on a piece of paper, on the gauze, on the steam plate. Cover in such a manner that no dust will enter, allowing the moisture to escape. Allow it to stand until a constant weight is obtained, which usually requires about one hour and a half. Remove, cool and weigh. Volatilize the mercurous chloride by the application of heat, cool and weigh. The difference in weight gives the amount of mercurous chloride. To convert the mercurous chloride to metallic mercury, use the factor .8496.

MERCURY (By Electrolysis)

Dissolve a 5 gram sample in 35 c. c. nitric acid; dilute to 225 c. c. and electrolyze with a weak current (.25 amperes, approximately)

The mercury is deposited slowly. The current is gradually increased until the mercury is all deposited. This sometimes takes three days, current being increased each day. Copper, if present, will be deposited with the metallic mercury. Weigh electrode, then distil off the mercury with a gentle heat. The loss will be mercury and the residue, copper.

Determination of Impurities in Spelter

Spelter is supposed to be virgin zinc and is therefore analyzed for impurities only:

COPPER AND LEAD

Dissolve 5 grams of the sample in 35 c. c. nitric acid and about 30 c. c. water in a 250 c. c. beaker and dilute to 225 c. c. Electrolyze over night for copper and lead. Lead is deposited as dioxide. Multiply by factor .8661 to obtain lead. Difference or increase in weight of negative electrode is weight of metallic copper.

IRON

Dissolve 5 grams of sample in 30 c. c. hydrochloric acid, 5 c. c. nitric acid, then boil and make strongly alkaline with ammonia; boil and filter. If any appreciable amount of iron is present, filter and wash with hot water and dissolve from filter with about 20 c. c. dilute sulphuric acid (1 part acid and 4 parts water). Run through a Jones reductor and titrate with a standard potassium permanganate solution.

CADMIUM

Put 25 grams of sample in a 500 c. c. beaker. Add 250 c. c. water and 55 c. c. of hydrochloric acid (Sp. Gr. 1.19) a little at a time and finally let stand over night. The following day add more acid with stirring, using about 2 c. c. at a time, and allowing to stand after each addition of acid until finally all but about 2 grams of zinc have been dissolved. About 60 c. c. of acid in all is usually required. Filter, first transferring one of the undissolved pieces of zinc to the filter. Wash with water and discard the filtrate. Transfer undissolved matter to a casserole, cover, and dissolve in nitric acid. Add 200 c. c. of 1:1 sulphuric acid and evaporate to white fumes. Take up with 100 c. c. water, boil, cool and filter off lead sulphate. Dilute filtrate to 400 c. c., add 10 grams ammonium chloride and pass hydrogen sulphide for one hour. It is occasionally necessary to start precipitation by the addition of a drop or two of ammonia. Let precipitate settle, filter impure cadmium sulphide in a loose bottomed Gooch, remove cadmium sulphide and asbestos, cleaning inside of crucible with a little asbestos pulp, add 60 c. c. of 1:5 sulphuric acid and boil one-half hour. In case of spelters carrying large amounts of cadmium it may be necessary to add more acid. Filter off undissolved lead sulphide, wash and dilute filtrate to 300 c. c. Add 5 grams ammonium chloride and reprecipitate with hydrogen sulphide to remove traces of zinc. In case of large amounts of cadmium a third precipitation may be necessary. After the final precipitation, filter and transfer to a weighed platinum dish, cover, and dissolve in 1:3 hydrochloric acid. Also dissolve the sulphide remaining on the filter paper in hot 1:3 hydrochloric acid and add it to the solution in the platinum dish. Add a little sulphuric acid and evaporate to white fumes. Cool, add 2 c. c. water and 1 c. c. nitric acid to oxidize any filter paper shreds and again evaporate to white fumes. Finally remove excess of sulphuric acid by heating dish cautiously at about 500° C. or to a dull redness. Weigh as cadmium sulphate.

$CdSO_4 \times .539 = Cd.$

NOTES: Instead of removing the bulk of the lead as sulphate and the balance of the lead and copper as sulphides, they may be electrolytically separated as follows:

Wash the undissolved particles of zinc and cadmium remaining on the filter from the original solution back into the original beaker, cover, and dissolve in 25 c. c. concentrated nitric acid. Dilute to 125 c. c. and electrolyze on unweighed electrodes with rotating cathode using a current of about 10 amperes. All traces of copper and lead will be deposited in 20 minutes.

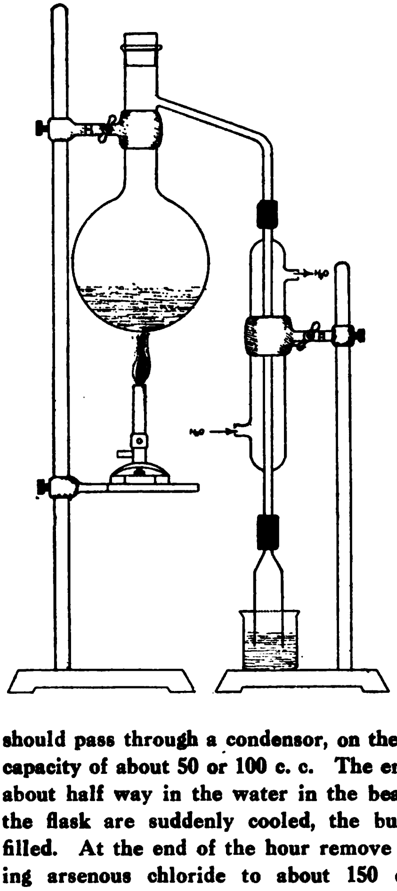
Electrolytic Precipitation of Cadmium—Instead of completely separating the cadmium from the zinc with hydrogen sulphide the cadmium can be deposited electrolytically from the first impure mixture of zinc and cadmium sulphides as follows:

Take the sulphuric acid filtrate from the solution of the first mixture of the zinc and cadmium sulphides to complete dryness. Dissolve the residue in 50 c. c. hot water and filter. Wash with hot water to 125 c. c. volume in 180 c. c. beaker, add 4 c. c. of 10 per cent sulphuric acid solution and 3 c. c. of a saturated solution of ammonium sulphate. Electrolyze on a platinum gauze with a current of .08 ampere over night. Four hours is sufficient for .1 per cent to .2 per cent of cadmium such as is usually found in spelter. No zinc will be deposited under these conditions even when a large excess of zinc sulphate is present.

Battery Zincs—Cadmium in battery zincs is determined in the above manner, but the mercury must be removed. In this case the hydrogen sulphide method of removing copper and lead is preferable, for it will also rmove mercury at the same time.

Method of Analysis of Pig Tin

ARSENIC



Dissolve 2 grams of ti sample in 10 c. c. concentrated sulphuric acid in a 25 c. c. Erlemeyer flask (Jena) Cool and transfer to a 50 c. c. distilling flask (as pe diagram) using as littl water as possible in transferring (not more than 10 o 15 c. c.) Now add 100 c. c concentrated hydrochlori acid (the hydrochloric aci can be used for transferring and 25 grams of ferrou sulphate, heat until th distillation starts, then boi slowly for thirty minutes Now add 100 c. c. mor hydrochloric acid and boi one-half hour longer, at the end of which time th arsenic will all be distilled Catch the distillate, which i hydrochloric acid and arsenous chloride, in a beaker o flask containing about 50 c.c of water. The distillate should pass through a condensor, on the end of which is a bulb with capacity of about 50 or 100 c. c. The end of the bulb should dip dow about half way in the water in the beaker so that if the contents o the flask are suddenly cooled, the bulb cannot be more than hal filled. At the end of the hour remove and heat the solution containing arsenous chloride to about 150 degrees Fahrenheit, and pas hydrogen sulphide gas through it for thirty minutes. Let it stan for at least ten hours. Now filter through a Gooch crucible, wash wit hot water, absolute alcohol, then with carbon bisulphide and finall with alcohol. Dry at 212 degrees Fahrenheit. Weigh as arseniou sulphide. After reaching a constant weight, dissolve out the arsenious sulphide with a warm solution of ammonium carbonate, (1:5) wash thoroughly with water and alcohol, dry and weigh. The loss i weight is arsenious sulphide.

ANTIMONY

Determine same as antimony in alloys, using a 1-gram sample. As the arsenic is determined along with the antimony in this titration method, a correction must be made, by deducting one and six-tenths (1.60) times the amount of arsenic found by the method given for arsenic.

LEAD AND COPPER

The lead and copper in pig tin are determined by dissolving a 1-gram sample in 15 c. c. nitric acid (1.42 Sp. Gr.) and 5 c. c. hydrogen peroxide and evaporating to dryness. Take up in 50 c. c. dilute nitric acid, (1 part acid to 4 parts water) warm, dilute to 250 c. c. and electrolyze over night for copper and lead.

Other impurities which may be present must be treated individually.

Method of Determining Tin and Lead in Tin and Terne Plate

The standard size of Tin and Terne Plate purchased under P. R. R. specifications is 28 x 20 inches. The sheet selected for analysis is cut crosswise at equal distances from the ends, and a strip two inches wide cut the full width of the sheet adjacent to this middle cut. This strip is examined for weight of plate and weight of coating.

METHOD OF DETERMINING WEIGHT OF PLATE

Weigh the strip cut from the middle of the sheet and calculate to pounds per square foot. Area of strip 20″ x 2″ equals 40 square inches. 144 square inches (1 square foot) divided by 40 equals 3.6. That is, there are 3.6 squares of 40 square inches each in one square foot. In one pound avoirdupois there are 453.6 grams. Therefore, weight of 40 square inches in grams x 3.6 divided by 453.6 equal weight per square foot in pounds This is equivalent to weight of 40 square inches in grams x .007936 equals weight per square foot in pounds.

APPARATUS AND REAGENTS

Some form of tin-smith's shears; 200 c. c. graduated flask; 25 c. c. and 50 c. c. pipettes; 100 c. c. burette; rubber stopper fitted with Bunsen valve; wash bottle; 250 c. c. Erlemeyer flask; lump marble; iodine solution approximately N/$_{10}$ hydrochloric acid; starch solution; and tank of carbon dioxide or apparatus for generating same.

METHOD OF DETERMINING TIN

The success of this method depends largely upon the degree of exclusion of air from contact with the stannous chloride solution. Details are as follows: From the center of the 20 inch strip cut a piece exactly two inches square, and then cut this piece into strips which will pass through the neck of the 200 c. c. graduated flask. In the meantime, immerse the wash bottle, filled with distilled water in a basin or any other receptacle containing ice water, and connect with the supply of carbon dioxide, allowing gas to bubble through the wash water for about 10 or 15 minutes. Also prepare the graduated flask by adding 100 c. c. concentrated hydrochloric acid and three pieces of marble about the size of a pea, inserting the Bunsen valve at once. By the time the marble is nearly dissolved the air will be driven from the flask, and the strips of tin should be dropped into the flask as quickly as possible and the Bunsen valve replaced. Stand on steam plate and when the solution is nearly completed drop in a small piece of marble, replace the stopper and cool at once under the tap and then in ice water. The use of the marble is to insure the presence of an inert gas in the flask at all times. When the solution is ice cold, remove the stopper and fill to the mark on the neck with the water saturated with carbon dioxide. Place a solid rubber stopper in the flask and shake well to dissolve all separated salts and obtain homogeneous mixture. Remove the solid stopper and replace with a two-hole stopper through one hole of which passes a small glass tube extending nearly to the bottom of the flask, and above the stopper fitted with a short piece of ⅛ inch diameter rubber tubing into which the small end of a pipette will fit tightly. Having previously fitted the burette with the standard iodine solution, insert the tip of the 25 c. c. pipette into the rubber tube and withdraw 25 c. c. of solution. Immerse the tip of the pipette below the surface of the iodine in the flask and allow to discharge. Add starch solution and complete the titration. Note the number of c. c. taken. For example the burette reading would be as follows: 15.2 c. c. of iodine solution used for the preliminary titration of the 25 c. c. taken. As 15.2 were required for the preliminary 25 c. c. titration; double this amount, viz: 30.4 c. c. of iodine solution were added to the flask before pipetting off the 50 c. c. To complete the titration .6 c. c. additional were required or a total of 31.0 c. c. Since we started with four square inches of plate and diluted the solution to 200 c. c., 50 c. c. would represent one square inch. Therefore, 31 (the number of c. c. used) x value of one c. c. of iodine solution equal grams tin in one square inch. To reduce grams per square inch to pounds per square foot multiply by the factor .317465, which is equivalent to multiplying the number of grams of tin per square inch × 144 divided by 453.6.

STANDARDIZING THE IODINE SOLUTION

Make up a stock solution by dissolving 26 grams of iodine and 36 grams of potassium iodide in a small amount of distilled water and dilute to 2 liters. For the approximately $N/_{40}$ solution, dilute 50 c. c. stock solution to 2 liters. This may be corrected to exactly $N/_{40}$ if desired. To standardize, weigh 4 samples of .1 gram each of C. P. Tin into a 250 c. c. Jena Erlemeyer flask, add 10 c. c. concentrated sulphuric acid and 5 grams of potassium sulphate. Dissolve over bare flame, holding with crucible tongs and at the same time agitating the contents to prevent any possible bumping or loss mechanically. The contents will at first be black, then whiten. Keep agitating over flame until the contents are white or very light gray and dense wh fumes of sulphuric anhydride come off freely. Let it cool, then add c. c. of a solution of tartaric acid containing 5 grams tartaric acid, 10 c. c. concentrated hydrochloric acid, boil for about three minut Cool down under water tap and add 130 c. c. of water and 10 c. c. c centrated hydrochloric acid. Cool down in ice and water and ad few drops of potassium permanganate solution to pink color, to disp of any antimony if present. Transfer this solution into a long n 500 c. c. Jena round bottom flask which is provided with a rub stopper and bent tube, with 50 c. c. concentrated hydrochloric a Add about 1 gram antimony, powdered flour fine, and place the fl into a bath of boiling water for about 15 minutes. Now conduc stream of carbon dioxide through the solution, allowing gas to fl gently and having the end of the outflow tube immersed in the water prevent air from drawing back and causing oxidation. Heat to b ing for five minutes over a bare flame then cool down rapidly in water, all the time having the carbon dioxide passing through solution. When cool, add starch solution and titrate quickly and n number c. c. taken.

Run a blank as above, using same amount of reagents and ded from number of c. c. found in above titration. Divide the weight the sample taken, by the number of c. c. remaining after correcti which gives the value of 1 c. c. of iodine solution in terms of tin.

DETERMINATION OF LEAD

Cut a piece two inches square from the center of plate same as the determination of tin. After cleaning with chloroform, weigh and it fine. Take 2 grams in a 250 c. c. beaker and dissolve on the ste table with a mixture of 10 c. c. concentrated sulphuric acid and 30 c of water, add water occasionally, if necessary, to prevent separation the iron salts. When the iron is all in solution, indicated by no furt evolution of gas, dilute to about 150 c. c. with water and filter thro a 7 c. m. filter paper washing two or three times by decantation w water acidified with sulphuric acid. The small amount of coat which may be adhering to the filter paper is washed back into beaker by means of an intermittent stream of water from a w bottle, and then 30 c. c. of nitric acid added (15 c. c. water and 15 c concentrated C. P. nitric acid) or the warm acid may be pou through the filter paper. Heat the solution to dissolve the lead, tin being precipitated as metastannic acid. Dilute to 200 c. c. a without filtering, electrolyze cold. The lead is deposited on the an as PbO_2. It is advisable to start the electrolysis with a current about 0.5 ampere and finish with 1.0 ampere. To wash the electro have three beakers of water ready, remove the beaker with the electrolyte and replace quickly with one of distilled water. Repeat this operation at least twice. Remove the electrolyte, dry and weigh. This weight × .8661 equals weight of lead.

Lead is reported in terms of (1), gms. per sq. in. of plate; or (2), lbs. per sq. ft. of plate; or (3), lbs. per 112 sheets 20 in. x 28 in.

Example:

PbO_2 from 2 gms. sample = 0.160 gms.
Wt. of 4 sq. in. of plate = 6.7707 gms.

(1). Gms. per sq. in. of plate.

$$\frac{PbO_2 \text{ from 2 gms. sample}}{2} \times .8661 \times \frac{\text{wt. of 4 sq. in. of plate}}{4} =$$

$$\frac{.160}{2} \times .8661 \times \frac{6.7707}{4} = .1172 = \text{grams per sq. in. of plate.}$$

(2). To reduce gms. per sq. in. of plate to lbs. per sq. ft. multiply by .3175. .1172 × .3175 = .0372 lbs. per sq. ft. of plate.

Or in a simpler form—

PbO_2 from 2 gms, sample × wt. of 4 sq. in. of plate × .03437 = lbs. per sq. ft. of plate. .160 × 6.7707 × .03437 = .0372 lbs. per sq. ft. of plate.

(3). To reduce gms. per sq. in. to wt. of coating in lbs. per 112 sheets 20 in. x 28 in., multiply by 138.3. 138.3 × .1172 = 16.2 lbs. per 112 sheets 20 in. x 28 in.

Or in a simpler form—

PbO_2 in 2 gms. sample × wt. of 4 sq. in. of plate × 14.95 = wt. of coating in lbs. per 112 sheets 20 in. x 28 in. .160 × 6.7707 × 14.95 = 16.2 lbs. per 112 sheets 20 in. x 28 in.

NOTE—Tin and terne plate are usually sold by the manufacturer on a basis of the wt. of a box of black sheets, and the weight of the same sheets after coating. The consumer is not interested in the wt. of heavy coating on certain portions of the sheet, known as "list edge" He is interested in the wt. of coating on the average surface, consequently the sample is taken from the surface representing an average surface.

CHECK METHOD FOR TIN

Take a sample two inches square as in the other method, cut into strips, place in a 500 c. c. Erlemeyer flask, add 40 c. c. water and 40 c. c. concentrated hydrochloric acid. Place on steam plate just long enough to start action. Remove and support in an inclined position as care must be taken to prevent loss of tin by volatilization. When in solution, filter into a 500 c. c. long neck, round bottom Jena flask, washing with a mixture of 80 c. c water and 20 c. c. hydrochloric acid. Place the flask in a hot water bath, add 1 gram of powdered antimony and proceed as in standardization of iodine solution.

Method of Determining Zinc Coating on Galvanized Sheets

The sample for determining the weight of plate and amount of coating should be taken from near the middle of the sheet. It should be exactly two inches square, and is best cut from the plate by means of a pair of shears, or ordinary tin-smith's shears.

Weigh carefully and place in a 250 c. c. beaker containing a platinum crucible cover, or piece of scrap platinum. The galvanized sheet should rest upon or come in contact with the platinum, which will hasten the solution. Add water to cover the plate, and the 5 c. c. of sulphuric acid, concentrated. Cover the beaker with a watch glass. In from ten to twenty minutes the zinc will be dissolved, indicated by the cessation of the rapid evolution of gas. The small amount now coming off is due to the action of the acid in the base plate and will come mainly from the edges.

Wash the plate with a jet of water, allowing the washings to run into the beaker. Wipe the plate dry and weigh.

Not only the zinc coating will go into solution, but also a portion of the iron plate. Immediately upon the removal of the plate from the sulphuric acid, titrate the solution with standard permanganate of potash and calculate the weight of the iron dissolved which should be deducted from the total loss of the weight of the plate. This gives the amount of zinc coating on four square inches of plate.

Weight of coating on four square inches divided by 4 × 144 equals grams per square foot, divided by 28.35 equals ounces per square foot. Or, weight of coating in grams on four square inches × 1.27 equals weight in ounces per square foot.

Example:	Weight of four square inches equals	17.0892 grams
	Weight after treatment	15.6991 grams
	Weight of zinc plus iron	1.3901 grams
	Weight of iron in titration	.0403 grams
	Weight of zinc coating	1.3498 grams

1.3498 × 1.27 = 1.71 ounces per square foot.

To obtain weight of plate per square foot multiply weight of four square inches in grams by 1.27 equals weight in ounces per square foot.

Determination of Sulphur in Brasses, Bronzes, Iron and Pig Iron

The Sulphur in the brass or bronze is precipitated as copper sulphide. This copper sulphide is oxidized with bromine to copper sulphate and the Sulphur determined by precipitation with barium chloride. The purpose of the CO_2 is to insure that all of the Sulphur remains on the filter as copper sulphide. This method removes the large bulk of the metals present thus insuring a pure $BaSO_4$ precipitate.

Dissolving solution—

Copper Potassium Chloride	650	grams
Water	2	liters
Hydrochloric Acid	1.5	c. c.

Put 300 c. c. of dissolving solution in each of three 500 c. c. Erlenmeyer flasks, connect these flasks in series so that CO_2 can be bubbled through the liquid in each of these flasks. Then add 10 c. c. of hydrochloric acid to each flask and place apparatus on the steam plate and pass a continuous stream of CO_2 for 30 minutes. Introduce the samples (1/3 grams) wrapped in 7-centimeter filter papers into the second and third flasks in the series as quickly as possible. Continue to pass CO_2 until all is in solution, shaking every 15 minutes. The first flask is the blank determination. Filter as rapidly as possible through a 9-centimeter filter and wash with CO_2 water twice. Return filter papers to the original flasks and add 25 c. c. bromine water and 5 c. c. hydrochloric acid. Cover with watch glasses and place on steam plate for 30 minutes. Remove covers and allow the fumes to escape. Add about 1/10 gram C. P. sodium carbonate and evaporate to dryness to render any silica insoluble. Take up with 5 c. c. hydrochloric acid, 3 grams ammonium chloride, and 50 c. c. water. Boil, filter, wash and determine Sulphur by precipitation with barium chloride in the usual manner. Run blank and calculate per cent Sulphur.

$BaSO_4 \times .1373 = S.$

Antimony

Determine Antimony as in the analysis of Lining Metal and Babbitt, Method No. 9. Any arsenic present will titrate as antimony and must be determined and subtracted.

ARSENIC

Determine as in Arsenic in Tin, Method No. 12.

LEAD AND COPPER

Determine as in Lining Metal and Babbitt, Method No. 9.

Antimony is usually brought under specification for special purposes. Its principal use is in making Babbitts, and hardened lead alloys. Different uses make it necessary to watch for different impurities, and the individual chemist must be guided by his particular need in his search for impurities.

Bismuth

Dissolve 1 gram of the alloy in 10 c. c. nitric acid (Sp. Gr. 1.42) and concentrate to pasty mass. Add 7 c. c. hydrochloric acid (Sp. Gr. 1.19) and heat until the alloy is decomposed or in solution. Add 8 c. c. sulphuric acid and heat to white fumes. Cool, add 30 c. c. of water and boil until all bismuth sulphate goes into solution. Cool, filter and wash with a 10% solution of sulphuric acid. Filter as soon as possible, otherwise some basic bismuth sulphate will filter out. Dilute to 100 c. c. and precipitate bismuth, copper, arsenic, antimony and traces of lead with hydrogen sulphide. Filter and wash with dilute hydrogen sulphide. Digest precipitate and filter in a concentrated solution of potassium cyanide using as little as possible to insure solution. Warm for a few minutes, filter and wash with hot water. Dissolve the sulphides of bismuth, lead and cadmium by heating the filter and precipitate with 10 c. c. of dilute nitric acid. Add 20 c. c. of water and filter, using a porcelain Gooch. Wash with dilute nitric acid. Transfer the solution to a 400 c. c. beaker, dilute to 300 c. c., and heat to boiling. Remove the burner and drop a bit of litmus paper into the hot solution and neutralize the solution with dilute ammonia until solution becomes slightly cloudy. Then add 1 c. c. of dilute hydrochloric acid and digest on the hat plate without boiling for one hour. Filter the precipitate of bismuth oxychloride on a porcelain Gooch; wash with hot water; dry at 100° C. and weigh.

$BiOCl \times .8017 = Bi.$

BLEACHING POWDER (CHLORINATED LIME)

Weigh accurately 7.090 grams of the sample and grind it to a thin paste with a little water in a mortar. Rinse into a 100 c. c. flask. Fill to the mark and mix thoroughly. (50 c. c. solution=.3545 grams of CaOCl.) Take 50 c. c. of this solution and run in alkaline sodium arsenite $N/_{10}$ which is made up as follows: Weigh carefully 4.95 grams of As_2O_3 into a 250 c. c. beaker and add 20 grams of sodium acid corbonate and boil until solution is complete. Rinse this into a gfiraduated liter flask and dilute up to the mark. 1 c. c. of this solution equals 1 per cent. available chlorine. 35 c. c., more or less, are usually required.

The end of the reaction is shown by aid of a piece of filter paper sensitized with potassium iodide and starch. When sufficient arsenite solution has been added, no blue color will develop when a drop of this solution is placed on the sensitized paper. (Potassium iodide and starch.)

Method of Determining Crystallized Ferrous Sulphate in Blue Vitriol

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Reduce the blue vitriol to small particles in a mortar, mix thoroughy and the into a 500 c. c. beaker weigh ten grams of this sample. Add 150 c. c. of water and dissolve, boiling the solution gently. When the solution is complete and while it is still hot, add carefully a few crystals of ammonium persulphate to oxidize the iron completely. Boil again for about five minutes. Now remove from the flame and make the solution strongly ammoniacal, adding ammonia until the precipitate of copper first formed is redissolved. Boil again a few minutes, then filter the solution through a carbon filter tube containing an asbestos pad. Wash the residue of ferric hydroxide on the pad several times with 1 to 1 ammonia water and finally with distilled water. Now dissolve the residue of ferric hydroxide on the pad with about 100 c. c. of 1 to 4 sulphuric acid, pouring about 75 c. c. through the pad several times and finally washing through with the remaining 25 c. c.

Now pass this solution through the Jones reductor and then wash with distilled water until about 700 c. c. of the solution is in the suction flask. Titrate this solution immediately with standard potassium permanganate solution whose iron value is known, and calculate the amount of iron in the sample. From the value of iron determined, calculate the equivalent value of crystallized sulphate. The Cubic Centimeters on $KMnO_4$ used × the iron factor of $KMnO_4$ solution divided by 2.014=the per cent. of $F_2SO_4.7H_2O$.

Asbestos Lagging

(Hydrated $MgCO_3$ and Asbestos)

Treat 5-10 grams with 20% $HC_2H_3O_2$. Heat until all is in solution except the asbestos. Now filter through a weighed 11 c. m. filter into a 500 c. c. or 1000 c. c. flask, cool and dilute up to mark. For analysis take c. c. containing .5 gram and determine SiO_2, Fe_2O_3, Al_2O_3, CaO and MgO. Dry the asbestos thoroughly and weigh. Ignite and weigh and get the per cent of dry and clean asbestos. Should be: 15% asbestos and 85% $MgCO_3$ hydrated.

Determine H_2O by placing a sample on the steam plate (212 F) to constant weight. Determine CO_2 by placing .5 gram sample in CO_2 apparatus and treating with 90 c. c. of a 20% solution of H_2SO_4, collecting CO_2 in KOH bulbs and weighing direct.

Report:—CO_2 as $MgCO_3$; excess Mg as MgO
H_2O as free and combined   For CaO—see Treadwell-Hall
and asbestos as such   For MgO—see Treadwell-Hall

Results of Analysis should be reported as given below:

15.77 %	Ignited Asbestos		
2.16 %	Loss on Ignition	17.93 %	Oven dried asbestos
.28 %	SiO_2		
.00 %	R_2O_3 (Fe_2O_3 & Al_2O_3)		
2.03 %	CaO		
34.22 %	MgO	81.78 %	Basic Carbonate of Magnesium
29.99 %	CO_2		
4.46 %	Water (moisture)		
10.95 %	Water (combined)		
99.86 %	Total		

For the combined water, burn a sample in a platinum crucible for several hours, cool and weigh. Calculate the % loss. From this deduct the % moisture, loss on ignition of asbestos (oven dried) and % CO_2. Example, wt. of loss .4756=47.56%—(29.99+4.46+2.16)= 10.95% combined water.

Method of Analysis of Caustic Soda or Lye

APPARATUS AND REAGENTS

Beakers, flasks, bottles, etc., as described under "Operation."

The solution of neutral barium chloride is made as follows:

Nearly fill a 16 or 18 liter bottle with distilled water and add 2 grams of C. P. barium chloride for every liter of water taken. Shake until dissolved. Add a little phenolphthalein for an indicator and then a saturated solution of barium hydroxide, a little at a time, until the solution is neutral, or very slightly alkaline, as indicated by a pink color. Allow it to stand over night for the precipitate, barium carbonate, to settle and then siphon off the clear solution as needed. The solution should be protected from the air by causing the air to bubble through caustic potash solution as it enters the bottle.

OPERATION

From the small stoppered bottle, weigh out 2 grams as quickly as possible and wash it into a graduated liter flask, using distilled water that has been freshly boiled to remove carbon dioxide. Stopper with a cork or rubber stopper and shake gently until all dissolved, then fill to the mark with a neutral solution of barium chloride prepared as directed below, while twirling. Stopper, shake thoroughly and let it stand several hours for the precipitate to settle, generally over night.

When the precipitate has settled, remove two portions of 100 c. c. each of the clear solution, using a pipette for the purpose. Titrate each portion with standardized HCl, using phenolphthalein for an indicator,

Method of Testing Amyl Acetate

TEST FOR MATERIAL INSOLUBLE IN SULPHURIC ACID

To 6 c. c. of amyl acetate in a graduated tube add enough sulphuric acid (1.84 Sp. Gr.) to make the entire volume 40 c. c. Cork and mix by repeatedly inverting the tube under cold, running water. Allow it to settle, read the volume of the separated insoluble layer and calculate the percentage.

Example: Assume the reading to be .4 c. c.:

(.4÷6)×100=6.66%) insoluble in sulphuric acid.

TEST FOR ACIDITY

Add 10 c. c. of amyl acetate to a beaker containing about 40 c. c. neutral alcohol, mix and titrate with $N/_2$ potassium hydroxide solution, using phenolphthalein as an indicator. Not more than .1 or .2 c. c. of the $N/_2$ potassium hydroxide solution should be necessary to produce the red end reaction, as the material is purchased on the supposition that it is nearly neutral.

TEST FOR MATERIAL INSOLUBLE IN WATER

To 10 c. c. of amyl acetate in a graduated tube add 10 c. c. of distilled water, shake to mix thoroughly, allow it to settle and read the volume of the insoluble layer. Subtract this from the 10 c. c. and calculate the remainder to percentage soluble in water.

Example: Assume that 9.6 c. c. were insoluble:

10—9.6=.4 and .4×100=4% material soluble in water.

DISTILLATION TEST

Distill 100 c. c. according the method used in analysis of Turpentine Substitute, Method 38. Compare the distillation values with those obtained from satisfactory amyl acetate, since the commercial amyl acetate is not C. P. normal amyl acetate.

SOLVENT POWER

Six grams of cellulose nitrate shall be dissolved in 100 c. c. of pure dry acetone. A like solution shall be made using the amyl acetate under examination as the solvent. A measured volume of each solution shall be taken and alcohol shall be slowly run into each solution from a burette. During the operation the mixing vessels shall be kept immersed and well shaken in a bath having a constant temperature of 25° C. The alcohol shall be added to these two solutions until a faint permanent opalescence is obtained in each vessel. The ratio between the volumes of alcohol required in the two solutions reckoned as percentage of that required in the pure acetone solution will represent the solvent power of the material under examination.

Method of Analysis of Sal Ammoniac

AMMONIA

Weigh .5 gram of the sample into an eight ounce Erlemeyer flask. Add 25 c. c. distilled water and when in solution, add 30 c. c. of half normal potash. Bring to gentle boiling and then set the flask, uncovered, on the hot plate for at least three hours. If the solution evaporates too much, add more water. Now when all the ammonia has been driven off, cool the flask and contents in an ice bath and when cold, titrate the excess of potash remaining in the flask with half normal sulphuric acid, using Methyl orange as an indicator. Run a blank the same way and at the same time as the sample, using exactly the same amount of potash. The number of c. c. of half normal sulphuric acid required to titrate the potash in the blank, minus the number of c. c. required to titrate the excess of potash remaining in the flask after all the ammonia has been driven off will give the number of c. c. of half normal potash required to displace all of the ammonia from 0.5 gram of ammonia chloride. This number of c. c. times 0.0085, divided by 0.5, times 100 will give the percentage of ammonia in the sample.

CHLORINE

Dissolve 0.5 grams in distilled water and titrate with standard silver nitrate solution, using potassium chromate as an indicator. Titrate to the first permanent appearance of red or brick red color due to the formation of silver chromate.

The silver nitrate is standardized against C. D. ammonium chloride from which the value of 1 c. c. in terms of chlorine is calculated.

The Volhard method for chlorides may be used:

The sample is dissolved in water and acidified with HNO_3. An excess of standard $AgNO_3$ is added and the $AgCl$ filtered off. The excess of $AgNO_3$ is titrated with standard KCNS using ferric alum as indicator. (See Treadwell-Hall, Vol. 1, page 707.)

Ash and sulphates may be determined if desirable.

Method for the Complete Analysis of Portland Cement

SILICA: Weigh a .5 gram sample of cement into a dry porcelain beaker (5 × 3 in.,) add 5 c. c. water and swirl at once to avoid settling of cement. When all is in suspension add 20 c. c. hydrochloric acid, specific gravity 1.15, (2 parts acid to 1 part water) and swirl until all is in solution (very little or no silica will separate this way.) Cover the beaker with a watch glass and triangle and place it on the hot plate. Evaporate to dryness.

When dry, dehydrate the silica by placing the beaker in an air bath, heated to 400° Fahrenheit for one hour The air bath is made so that the porcelain beaker is inserted in an opening in an asbestos board cover, about two-thirds of beaker being in bath, and supported by a clay ring so that it will not touch the metal bottom of the bath After dehydrating one hour, cool the beaker and add 30 c. c. of hydrochloric acid, specific gravity 1.15, heat to boiling, filter into a beaker and save the filtrate for iron oxide and alumina Wash the precipitate with a 2 per cent. solution of hydrochloric acid and hot water, ignite in a platinum crucible and weigh. Heat the contents in the crucible with water and hydrofluoric acid and a drop of sulphuric acid, evaporate to dryness, ignite, cool and weigh. Loss in weight equals silica.

The residue in the crucible may contain alumina, iron oxide, magnesia or lime, or a mixture of any or all of them, but is chiefly alumina and ferric oxide and may be added to the amount of alumina and ferric oxide obtained later, provided it is not too large. The residue should be tested for alumina, iron, magnesium and calcium.

ALUMINA AND IRON OXIDES:—Place the filtrate from the silica determination into a casserole, add ammonia in slight excess, boil gently to remove excess of ammonia. Allow the precipitate to settle when the odor of the solution indicates that the excess of ammonia has been reduced to the proper point, filter on a 9 c. m. filter paper, into a 400 c. c. beaker and wash with hot water. Wash the precipitate back into the casserole, being careful to keep the filter intact. Add 5 c. c. hydrochloric acid, specific gravity 1.20. Do not heat until solution is about complete. Add ammonia slightly in excess, and again drive off the excess of ammonia as previously described, by heating. Filter, using same filter and allowing filtrate to be collected in the original beaker. Ignite the precipitate and weigh the alumina and ferric oxide.

LIME:—To the combined filtrates which contain the lime and magnesia add 10 c. c. of ammonia, specific gravity 0.90, and bring to boil; add 25 c. c. of 4 per cent. ammonium oxalate solution, place a glass rod in the beaker to prevent bumping, cover, and boil for 5 minutes. Allow it to settle for one hour, filter and wash with hot water. Save the filtrate for magnesia. Ignite the precipitate in a platinum crucible over a Chaddock burner surrounded by clay ring to constant weight and weigh as lime (CaO.)

MAGNESIA:—Transfer the filtrate from the calcium oxalate to a porcelain casserole; add 50 c. c. nitric acid, specific gravity 1.42, to destroy ammonia salts. Cover the casserole with a watch glass on a triangle and evaporate to dryness on the steam plate. Add 12 to 15 drops of hydrochloric acid, specific gravity 1.15, to the residue in the casserole, evaporate to dryness; take up with 10 to 12 drops of hydrochloric acid, specific gravity 1.15, and a little water Add ammonia in excess and a few drops of a solution of ammonium oxalate, boil and filter into a 200 c. c. beaker. If the calcium oxalate previously determined did not run through the filter this residue here filtered off is due to dirt, etc., and is discarded. If it is found that lime is present in the residue it is determined and added to the previous result in order to obtain the total lime (CaO). To the filtrate containing the magnesia add 10 c. c. of a saturated solution of microcosmic salt. Cool in an ice bath and add 10 c. c. ammonia, specific gravity 0.90, with constant stirring. Allow it to stand over night, filter and wash the precipitate with a 10 per cent. solution of ammonia. Wash finally with an ammoniacal nitrate of ammonia solution (30 c. c. ammonia, 60 c. c. water and 10 grams ammonium nitrate,) dry, ignite with the usual precautions, and weigh as magnesium pyrophosphate. Dissolve the precipitate in a dilute solution of hydrochloric acid (1 part acid to 1 part water,) filter, wash with water, dry, ignite and weigh. Deduct this weight from the weight of the magnesium pyrophosphate. Use the factor .3624 to convert the magnesium pyrophosphate to magnesia.

LOSS ON IGNITION:—Ignite 0.5 gram of sample in a platinum crucible surrounded by clay rings on a Chaddock burner for one-half hour. Do not use blast. Cool and weigh.

ALKALIES:—The alkalies are determined by the J. Lawrence Smith method.

ANHYDROUS SULPHURIC ACID:—Weigh into a casserole or 250 c. c. beaker a 2-gram sample of cement. Suspend with water from a wash bottle, using 10 to 20 c. c. of water, and dissolve with 20 c. c. hydrochloric acid (Sp. Gr. 1.15). Boil about 10 minutes. Transfer solution to a 500 c. c. graduated flask, which contains 20 c. c. concentrated ammonia and 50 c. c. water. Mix the solution thoroughly. Solution should be ammoniacal. Dilute to mark, mix well and allow it to settle. The solution should be at room temperature. Filter through a dry filter paper exactly 250 c. c. of solution into a 250 c. c. graduated flask, which will now represent 1 gram of cement sample. Transfer this to a 500 c. c. beaker, add hydrochloric acid in slight excess. Heat to boiling and precipitate with 15 c. c. of a low per cent. barium chloride solution. Continue boiling about 15 minutes; allow the barium sulphate precipitate to settle completely, filter, ignite and weigh.

Wt. Barium Sulphate × .3429 ÷ 1 × 100 equals percentage of Anhydrous Sulphuric Acid in the cement.

MAGNESIA

Alternative Method for Magnesium:

Into a 700 c. c. casserole weigh a 2 gram sample of cement. Add about 50 c. c. water and 50 c. c. concentrated hydrochloric acid, C. P., and cover with a watch glass. Heat over a bare flame until solution is complete. Add 75 c. c. of ammonium oxalate solution (112 grams ammonium oxalate to 1 liter water). Heat to boiling again. Remove the casserole from the flame and add slowly a solution of 80 c. c. of ammonia, C. P., (0.90 Sp. Gr.) and 20 c. c. water. Boil about 2 minutes and transfer to a liter flask, diluting with water to the liter mark, and mix thoroughly. Allow to stand over night for the precipitate to settle completely.

Filter off 500 c. c. of the clear liquid for the determination of the magnesia. This may be done as follows: To a suction flask fit a carbon tube in which is placed a pad of asbestos; to the top of the carbon tube, which is provided with a perforated rubber stopper, is attached one end of a bent glass tube, the other end of which extends nearly to the bottom of the liter flask. When suction is applied to the suction flask, the liquid is transferred to this flask, passing through the asbestos filter which retains any sediment that may be carried over. A little more than 500 c. c. is filtered over, 500 c. c. is measured off and transferred to a liter beaker. Enough hydrochloric acid, C. P., is added to make the solution distinctly acid, the solution is cooled in ice and 15 c. c. of saturated solution of microcosmic salt (sodium-ammonium-phosphate) and 80 c. c. of C. P. ammonia (0.90 Sp. Gr.) are added. The ammonia is added slowly while the solution is agitated by stirring. When the ammonia has all been added the solution is stirred vigorously until the precipitate begins to form.

The solution is allowed to stand over night to insure complete separation of the magnesia. The precipitated ammonium-magnesium-phosphate is now filtered off, using a Gooch crucible and applying suction. The precipitate is washed with a 10 per cent. solution of ammonia. The precipitate is cautiously dried and finally ignited strongly over a bare flame, cooled and weighed.

The percentage of magnesia is calculated as follows:

Wt. Ppt.×.3624÷1×100=per cent. magnesia in the sample.

NOTE: A 2-gram sample was taken of solution and diluted to 1 liter. As only 500 c. c. of this solution was used the results are calculated on a 1-gram sample.

Beeswax

The purity of beeswax may be established by determining its melting point, its specific gravity and its saponification equivalent.

The melting point of pure wax is about 60° C., while that of paraffine, its chief adulterant, is from 52 to 55° C.

The saponification equivalent of beeswax is from 88 to 107, while that of paraffin is 0.

The specific gravity of beeswax is approximately .969, while the specific gravity of paraffin is approximately .871.

The method of determining the specific gravity of beeswax is as follows:

Place a weighed rod of the wax, about 1 to 1.5 cm. long by 0.5 cm. diameter, in an accurately marked 50 c. c. flask, and run in water from a burette till the water level reaches the mark. Fifty c. c. minus the burette reading represent the volume occupied by the wax. The rod should be made to lie flat on the bottom of the flask, so that the incoming water will force its end against the sides and prevent the end from rising above the mark. The weight of the rod, divided by its volume gives its specific gravity.

The melting point is determined by putting small shavings of the sample in a capillary tube and heating this tube in a water bath with a thermometer according to the ordinary method of determining melting points.

The saponification equivalent is determined as in the analysis of oils.

Fiber Conduit

1. The walls of the conduit shall be compact and incapable of separation into layers after an immersion for 1 hour in boiling water.

2. The conduit shall in no way be affected by digestion in 2 per cent. phosphoric acid or 2 per cent. lime water for 48 hours at room temperature.

3. A 6-inch sample shall be thoroughly dried at a temperature of 110 degrees F. for 4 hours, then weighed, and after an immersion in water for 48 hours at room temperature, removed, dried with a towel, shall show no more than 4 per cent. increase in weight due to absorption of water.

4. A sample of conduit obtained by crushing a length and having an area of approximately 3 by 4 inches, with unfinished edges, when held over a burning match and ignited, shall not continue to burn any appreciable length of time after the match has been removed. The sample shall be held in a horizontal position with the concave side down, the match being applied to the under side and in the still air.

5. Acetic acid and Acetates: Break up with a hammer or make 30 or 40 grams of conduit sawdust. Place in a 1000 c. c. beaker and add 500 c. c. of benzol. Allow the beaker to stand 6 or 8 hours. Pour off the liquid, taking care not to allow any pieces of conduit to be poured out. Place this beaker on the steam plate on a piece of paper until all benzol has been evaporated off. Repeat the treatment of the first beaker, with 500 c. c. more benzol, and allow this to stand for several hours. Pour this into the second beaker and again evaporate off the benzol. When the last traces of benzol are being driven off, remove the beaker and pour its contents into a distilling flask. Add 6 c. c. of water, plus 1 gram of chemically pure phosphoric acid. Distill the mixture rapidly up to 170 degrees Centigrade and collect the distillate in a test tube. If the spattering cannot be prevented in the usual manner and some of the oil is carried over into the test tube, another distillation must be made of the contents of the test tube in a distilling flask. Transfer the distillate into a small separatory funnel, separate and draw off the water. Add 5 c. c. of standard alkali litmus solution to the water distillate. Should the latter cause the litmus to turn red, acetic acid or acetates are present in the conduit.

Method of Analysis of Sulphuric Acid to be Used in Making Electrolyte, and of Electrolyte

Determine the specific gravity of the sample at 60 degrees Fahrenheit, by means of a Westphal balance, as described in Method of Determining Specific Gravity of Oils and Other Liquids.

Note the color of the electrolyte or of the sulphuric acid diluted with distilled water so that its specific gravity is 1.20. Make all the following tests on the diluted material, which has been adjusted to 1.20 specific gravity.

QUALITATIVE TEST FOR HEAVY METALS

Take about 100 c. c. of the electrolyte in a 500 c. c. Erlemeyer flask; dilute with 50 c. c. distilled water, and add ammonia until the solution is only faintly acid. Pass into the warmed solution, hydrogen sulphide gas for 30 minutes, and note if a precipitate of the sulphides of the heavy metals is present. If such precipitate is found, filter and examine for such metals as mercury, copper, arsenic, tin, lead, etc., by the ordinary qualitative methods.

If more than a trace of any of these metals is present, determine the amounts by the ordinary analytical methods

IRON: To 10 c c. of electrolyte in a test tube, add an excess of ammonia, and note if more than a trace of iron is present. If an appreciable quantity is found, determine it as follows:

Take 100 c. c. of electrolyte at 60 degrees Fahrenheit and add a few c. c. of nitric acid, boil and add C. P. ammonia in strong excess. Boil until the precipitate of ferric hydroxide is well formed. Filter the solution through a carbon filter tube, having an asbestos pad which has previously been washed free from iron with acid. Wash with ammonia water (1 to 1) and finally with distilled water. Dissolve the residue from the pad with 100 c. c. of C. P. sulphuric acid (1 to 4.) Pass this solution through a Jones reductor, washing until 500 c. c. of the solution is in the flask. Titrate this solution at once with potassium permanganate, which has been standardized for iron. The number of c. c. of permanganate used, multiplied by the value of 1 c. c. of permanganate in terms of iron, gives the grams of iron in 100 c. c. of electrolyte. Calculate the weight in grams of 100 c. c. of electrolyte by multiplying its specific gravity at 60 degrees Fahrenheit by 100. The grams of iron found divided by the weight in grams of 100 c. c. of electrolyte, and multiplied by 100, gives the percentage of iron in the sample.

CHLORINE: To 100 c. c. of the electrolyte in a test tube, add 1 c. c. of silver nitrate reagent. Note if chlorine is present, and if in an appreciable quantity, compare it with the standard electrolyte containing the highest allowable percentage of chlorine, as follows:

Prepare a solution of ammonium chloride (C. P.) by dissolving .0181 gram in 100 c. c. of distilled water. Add to 1 c. c. of this solution in a test tube, 7 c. c. of water and 2 c. c. of sulphuric acid (C. P., 1.84 Sp. Gr.) thus making a solution of sulphuric acid of nearly 1.2 gravity, containing the highest allowable percentage (.001%) of chlorine. Add to the standard solution about 5 drops of silver nitrate reagent, and heat to boiling. Cool and pour into a 100 c. c. Nessler tube. Take 10 c. c. of the electrolyte sample (adjusted to 1.2 gravity.) Add 5 drops of silver nitrate reagent. Heat to boiling. Cool and pour into another 100 c. c. Nessler tube. Dilute the solution in each tube to the same volume, mix thoroughly and compare the intensity of the precipitate. If the precipitate of the sample is less than that of the standard, the electrolyte is accepted. If it is heavier than that of the standard, the chlorine must be determined before rejecting the sample. Determine the chlorine as follows:

Dilute 50 c. c. of the electrolyte to about 150 c. c. Add a few c. c. of strong nitric acid. Heat the solution to about 140 degrees Fahrenheit, and add silver nitrate reagent, drop by drop, with constant stirring until no more precipitate is formed. Heat nearly to boiling, stirring the solution vigorously until the precipitate coagulates, leaving a clear supernatant liquid. Failure to secure this result may be due to the absence of an excess of silver nitrate. Filter through a weighed Gooch crucible, and wash with hot water containing a little silver nitrate, which reduces very much the solubility of silver chlorine in water, and wash out the excess of silver nitrate with as little distilled water as possible Dry the crucible and contents on the hot plate or in an air bath, at about 300 degrees Fahrenheit, protecting it from the light as much as possible Cool and weigh. The increase in weight is silver chloride, which, multiplied by 0.2474, gives the weight of chlorine in grams in 50 c. c. of electrolyte, which divided by the weight of 50 c. c. of electrolyte in grams and multiplied by 100, gives the percentage of chlorine in the electrolyte. The weight of the electrolyte in grams is obtained by multiplying the number of c. c. taken by the specific gravity of the electrolyte.

MANGANESE: To 10 c. c. of the electrolyte in a test tube, add 1 c. c. of silver nitrate reagent, heat to boiling and add a few crystals of ammonium persulphate. Note if any manganese is present by the formation of the pink or purple permanganate acid. If an appreciable quantity is present, the following color method of comparison is used to determine approximately if the manganese content is higher than the allowed percentage (0.001.) Weigh into a 150 c. c. beaker accurately .05 gram of a standard steel containing 0.6 per cent. of manganese. Add 15 c. c. of silver nitrate solution, which is as follows:

2 grams Silver Nitrate
400 c. c. of water (distilled)
200 c. c. of Nitric Acid (C. P., Sp. Gr. 1.42)

Keep this silver nitrate reagent in a dark bottle.

Heat gently until solution is complete. Dilute with 50 c. c. of distilled water, bring to a boil, and add 15 c. c. of freshly prepared ammonium persulphate solution (100 grams of ammonium persulphate to 500 c. c. of distilled water.) Boil at least 5 minutes to bring out all the color of the permanganic acid. Cool and transfer it to one of a pair of comparison tubes, dilute the solution to exactly 100 c. c. volume.

Thirty grams of the electrolyte sample are taken for the comparison. Dividing the specific gravity of the electrolyte into 30 will give the equivalent number of c. c. To this number of c. c. add 15 c. c. of the silver nitrate solution. Boil, add 15 c. c. of the ammonium persulphate solution. Boil at least 5 minutes, cool and transfer to the other comparison tube; diluting to exactly 100 c. c., and compare the color with that of the steel solution. As before stated, this comparison is only an approximate method for determining the amount of manganese. Note if the color of the electrolyte solution is very appreciably lighter than that of the steel solution. It has been found unnecessary to determine the percentage of manganese in such cases as actual determinations show that it is less than that allowed. If, however, the shades of the colors of both the electrolyte and steel solutions are nearly the same, or if the electrolyte solution is deeper in shade, no dependence can be placed on the color comparison method to determine if the manganese content is higher or lower than the allowed amount. In such cases a modification of Marshall's method of titration of the permanganic acid with sodium arsenite has been found to give the best results. This method is as follows:

TITRATION OF MANGANESE (MODIFICATION OF MARSHALL'S METHOD)

Dissolve .3366 grams of arsenous acid with 1 gram of sodium carbonate in about 200 c. c. of distilled water, and boil off completely the carbon dioxide. Dilute largely with distilled water, and 8.5 c. c. concentrated nitric acid, and make the total volume 1000 c. c. One c. c. of this solution is about equivalent to .0001 grams of manganese. Standardize the solution as follows: Weigh 1 gram of a standard manganese steel into an Erlemeyer flask, and add 15 c. c. of the silver nitrate solution. Heat gently until the solution is complete. Dilute with about 50 c. c. of distilled water, bring to a boil and add slowly 15 c. c. of the freshly prepared ammonium persulphate solution. Boil about 5 minutes, allow to stand and cool for 10 or 15 minutes, cool in running water and titrate with the sodium arsenite solution, dividing the number of c. c. of the sodium arsenite solution required for the titration into the amount of manganese in the steel taken, gives the value of 1 c. c. of the solution in grams. The end point is reached when all the pink color of the permanganic acid has disappeared and a faint green color appears. In titrating manganese in electrolyte it has been found necessary to first neutralize the electrolyte with a solution of sodium hydroxide, as it was impossible to get concordant results by titrating the permanganic acid formed in the presence of a large amount of free sulphuric acid. By neutralizing the solution of electrolyte with sodium hydroxide, however, this difficulty was overcome, and close checks of a known amount of manganese in 1.24 specific gravity sulphuric acid have been obtained. The process applied to electrolyte is as follows: To 50 c. c. of the electrolyte add a rather concentrated solution of sodium hydroxide until the solution is just alkaline. Add 15 c. c. of the silver nitrate solution. The solution should now be acid (a faint cloudiness does not interfere with the results.) Bring to a boil, add slowly 15 c. c. of the ammonium persulphate solution. Boil about 5 minutes. Allow to stand to color and cool in running water. Be sure not to cool too soon, but allow sufficient time for all color to form. Titrate the solution to the disappearance of the pink color. It is necessary to titrate rather rapidly to the end point, as the color reappears rather quickly.

Multiplying the number of c. c. of sodium arsenite solution used by the volume of 1 c. c. of the solution in grams of manganese gives grams of manganese in the 50 c. c. of the electrolyte. Dividing the this result by the weight of 50 c. c. of electrolyte (found by multiplying 50 by the specific gravity) and multiplying by 100 gives the percentage of manganese in the electrolyte.

NITRATES: Mix 10 c. c. of the electrolyte and an equal volume of sulphuric acid (C. P., 1.84 Sp. Gr.) in a test tube, and when cool add carefully to the top of the solution without mixing 5 c. c. of a concentrated solution of ferrous sulphate. If a brown ring forms, it is an indication of the presence of nitrates. Nitrogen is estimated when necessary by the Kjeldahl method.

P. R. R. Specification No. 21 for Storage Battery Electrolyte specifics:

Sp. Gr.	1.200
Iron (maximum)	.005%
Chlorine (Maximum)	.001%
Nitrogen (maximum)	.005%
Copper (maximum)	.002%
Arsenic (maximum)	.001%
Magnesia (maximum)	.001%
Other Impurities	.010%

One hundred c. c. (Sp. Gr. 1.200 acid) must not require over .3 c. c. $N/_{10}$ $KMnO_4$.

Must be water white.

Method for the Determination of Formaldehyde in a Formaldehyde Solution—"Formaline"

(Modification of the Romijn Method)

PRINCIPLE:—Formaldehyde is quantitatively oxidized to formic acid by remaining in contact with iodine for a short time in an alkaline solution. $HCHO+H_2O+I_2=2HI+HCOOH$. 1000 c. c. normal iodine solution=HCHO divided by d,($\frac{30.02}{2}$)=15.01 grams formaldehyde. "Formaline" usually contains 40 per cent. of formaldehyde.

First determine the specific gravity of the solution with a Westphal balance and use 10 c. c. by putting it in a 250 c. c. graduated flask. Fill the flask to the mark with water. Now to a 250 c. c. Erlemeyer flask add 50 c. c. of an iodine solution from a pipette and follow this with 5 c. c. of the weak formaldehyde from the graduated flask (10 c. c. divided by $\frac{250}{5}$ =.2 c. c. of the original material). Immediately make alkaline by adding 10 c. c. of 13° Beaume sodium hydrate solution (10 c. c.=41 c. c. $N/_2$ HCl, approximately). Run a blank by using exactly the same amount of iodine solution. Allow to stand, shaking several times.

After 15 minutes add to blank and determination 10 c. c. of HCl (1-1.) Now titrate the excess of iodine with $N/_{10}$ sodium thiosulphate solution; when the dark color disappears and the yellow turns to a straw color add a few cubic centimeters of freshly prepared starch solution and titrate until the solution becomes water white. Subtract the number of cubic centimeters of $N/_{10}$ sodium thiosulphate solution used for the determination from the number used in the blank and multiply by the factor strength of the sodium thiosulphate in terms of formaldehyde, which theoretically should be .001501, divided by the weight of the sample taken and multiply by 100.

The $N/_{10}$ sodium thiosulphate solution is made by dissolving 24.8 grams of C. P. sodium thiosulphate in water and diluting to 1000 c. c. The exact strength of this solution may be determined from the iodine used in the Alloy Department, which is calculated in terms of tin.

The iodine used in the determination is an approximate $N/_1$ solution made by dissolving 26 grams iodine and 36 grams of potassium iodide in 25 c. c. water and diluting to 1400 c. c.

EXAMPLE: 71.05 $N/_{10}$ sodium thiosulphate solution is used in the blank, minus 16.75 c. c. used in the determination=54.30 c. c. Multiply this by .00149 (factor of $N/_{10}$ sodium thiosulphate in terms of formaldehyde.) Divide this by .2094 (10 c. c.×1.047 Sp. Gr. of formaline divided by $\frac{250}{5}$) and multiply by 100 per cent.=38.63 per cent. of formaldehyde.

Method of Determining Calcium Oxide and Calcium Hydroxide in Lime

Add sufficient freshly boiled, distilled water (30 c. c.) to 10 grams of lime in a mortar to form a thick milk. Add an excess of pure cane sgar solution and mix it intimately with the lime which forms a soluble saccharate. The sugar solution is made by dissolving 400 grams of cane sugar in a litre of boiling water. Transfer the milk of lime and the residue to a 100 c. c. flask, using the sugar solution of the above composition to wash the last portions from the mortar and to complete the volume to 100 c. c. Cool, dilute to the milk, mix and filter a portion of the mixture. Titrate 10 c. c. of the filtrate with normal hydrochloric acid, using phenolphthalein as indicator.

c. c. × 2.8 = per cent. CaO.

Method of Examining Fusees

Examine the fusees for the physical characteristics noting the diameter, the strength under load, the color of the paper, the printed directions for using, the security with which wooden plug and spike is attached, the construction of the head and how it is protected from moisture, the construction of the cap and the protection it affords, and protection given to the striking composition or scratch.

The strength, under load, is obtained by means of an ordinary hand operated tensile test machine of 600 pounds capacity. An iron bar is clamped in the upper jaws of the machine; this is fitted with two hooks or rings 10½ inches apart, which act as supports for the fusee. A third hook or ring is clamped in the lower jaws of the machine, by means of which the load is applied. In testing a five minute fusee, another bar with the hooks or rings six inches apart is used.

Place the fusee so that the hook comes as near the center of the composition part of the fusee as possible. Apply the power until the fusee breaks, noting the pounds required, which are registered on the dial.

To test the security with which the wooden plug and spikes are attached to the fusee, clamp plug in one jaw of the same tensile test machine and the other end of the fusee in the second jaw. Apply the power and note the pounds registered as in the strength under load test.

To determine if the head is protected from moisture, immerse the head of the fusee with the cap intact in water for five minutes, allow to drain for five minutes, remove the cap and examine it carefully to ascertain if any moisture has penetrated.

To determine the danger from spontaneous ignition; take the fusee after the above test and wrap it for about five inches, including the head composition, with three thicknesses of filter paper, moisten the paper and place the fusee in a steam heated oven which is kept at 100 degrees Centigrade for fourty-eight hours consecutively.

To test for ammonia salts, place a portion of the contents of the fusee tube in a beaker of water, add sodium hydroxide solution and note if ammonia vapors are present, detecting them by the odor.

Examine the fusees for their burning qualities according to the directions given in Specifications for Fusees.

Analysis of Fusees

HCl insoluble (Sulphur and Sawdust)
Bases—Ba, Ca, Sr, Mg, Na, K.
Red Fusee contains no Barium (or only trace)
Green Fusee contains Barium (no Sr or only trace)

Magnesium is not supposed to be present in either one, so only small amounts will be dealt with.

Owing to the fact that the ingredients of fusees are coarse and not well mixed, probably it is best to take the whole interior of the fusee, mixing well, and selecting from the quartered down sample, 10 grams for the bases, and 10 grams for the acids. Diluting to 500 c. c. and using 50 c. c. portions for analysis, representing 1 gram of the original sample.

Procedure: Weigh up, of the carefully prepared sample, 10 grams and place in a 300 c. c. beaker. Add 100 c. c. (1.4) HCl, boil 10 or 15 minutes and filter through a weighed porcelain Gooch (or counter balanced filter paper), wash with 200 c. c. boiling water to be sure of getting all the water soluble materials from the residue. Transfer the filtrate in the suction flask to a 500 c. c. graduated flask, cool and dilute to the mark and mix well. Label this flask "A".

The residue in the Gooch is dried on the steam plate for two hours, cooled and weighed. Attach the Gooch to a reflux condenser, by means of an aluminum or platinum wire, just below the lower end of the condenser arranged in a wide mouth 300 c. c. Erlemeyer flask, weighed, add 50 c. c. CS_2, warm gently until CS_2 begins to boil. Keep at this temperature for two hours. Remove the condenser and crucible, disconnect the crucible from the condenser, dry by suction and weigh to a constant weight. (5 hrs. at 95-100 C.) The flask containing the CS_2 is placed on a piece of asbestos on the steam plate and evaporated to dryness, weighed. This gives a check on the loss in wt. of crucible. Note: (Attention is called to the fact that the vapors of CS_2 ignite at temperatures far below a red heat, hence there is danger in placing the crucibles wet with the solvent in the drying oven immediately after extraction.) Ignite the crucible and cool, weigh, wt. of residue is ash. Usually about .1 to .5 of 1%.

Solution "A": for Ca, Ba, Sr, Mg, Na, and K. Unless the fusee is green it is unnecessary to look for Barium.

Procedure: Pipette out 50 c. c. of Sol. "A", run into a 250 c. c. beaker, add 30 c. c. of ammonium oxalate solution, heat to boiling and make ammoniacal and continue to boil 10 minutes. Set aside for 4 hours, when the oxalates will settle out from the sol., pour the clear liquid through a filter paper, cover the pptes. in the beaker with hot water containing a little ammonium oxalate. Treat the ppte. on the paper with hot HCl (1:4) collecting the resulting solution in a 250 c. c. beaker, wash paper well, reppte. Ca and Sr., adding the filtrate to that of the first precipitation. Label Filtrate "1".

Separation of Ca and Sr: Ignite the two oxalates and weigh as CaO and SrO, add 2 c. c. water to the crucible and enough HNO_3 to complete solution. Transfer to a 180 c. c. weighed Erlemeyer flask and evaporate to dryness. Place in an air bath and dry at 140-160 degrees C. for 2 hours. Cool and weigh. Treat the combined nitrates with 10 times its weight of absolute alcohol, allow it to stand 2 hours, then add its weight of ether, shake well and allow it to stand 12 hours or over night. It is then filtered through a 7 c. m. filter paper moistened with alcohol and ether, washed with alcohol-ether until a drop evaporated on a piece of Pt foil leaves no residue. The filtrate is evaporated to dryness in a luke warm water bath, the calcium nitrate is dissolved in water; ppte. as an oxalate, and after ignition, weighed as the oxide.

The residue in the flask is dried to remove alcohol-ether, taken up in warm water, transferred to a 250 c. c. beaker, its volume of alcohol added and a few drops of H_2SO_4. The Strontium is ppted. as $SrSO_4$. Allow it to stand over night, filter through a close Gooch, ignite to a dull red and weigh as $SrSO_4$.

Filtrate 1 for Mg. It is evaporated to dryness, ignited in a porcelain dish, the residue dissolved in water and dilute HCl. The carbonaceous matter if filtered off. The neutral solution containing only Mg and the alkalies (lithium must not be present), preferably in the form of chlorides is treated with an equal volume of 95% alcohol and then an excess of ammonium carbonate reagent. (180 c. c. conc. ammonium and 800 c. c. H_2O and 900 c. c. 95% alcohol and saturation with ammonium carbonate powder). The liquid is vigorously stirred for a few minutes and allowed to stand at least a half hour whereupon it is filtered through a Gooch crucible. The ppte. is washed with the precipitant, dried, ignited, and weighed as MgO. If considerable alkalies are present, the ppte. is dissolved in HCl, evaporated to dryness, taken up in water and the precipitation repeated. The combined filtrates are evaporated to dryness, treated with HCl, ammonium salts driven off and Na and K determined as below:—

Sodium and Potassium as $(Na\ and\ K)_2\ PtCl_6$. The HCl and NaCl is filtered to remove all carbonaceous residue, evaporated to dryness and weighed as chlorides, the chlorides dissolved in water. The assumption is made that the combined weight of the 2 Pt salts is NaCl, and on this basis the amount of chlorplatinate to be added can be calculated.

The reaction may be written:

$$\underbrace{2\ NaCl}_{116.92} + \underbrace{H_2\ PtCl_6}_{195.2} = Na_2\ PtCl_6 + 2\ HCl$$

From this reaction it is seen that 1 gram NaCl requires

$$\frac{195.2}{116.92} = 1.67 \text{ gm. Pt.}$$

Since our reagent contains 5 gms. Pt. per 100 c. c., 1 gram will require 20 × 1.67 = 33.4 c. c. of reagent. Therefore multiply the number grams of salt by 33.4 to obtain the c. c. of reagent.

A few tenths of a c. c. more than necessary is added and the whole evaporated nearly to dryness on a water bath at as low a temperature as possible. After cooling, the residue is treated with a few c. c. of abs. alcohol. Stand it under a bell jar for a few hours, remove and break up the mass with a stirring rod. The liquid is then decanted through a filter paper moistened with alcohol, and the treatment of the residue with alcohol, together with the breaking up of the powder, etc., is repeated, until the alcohol runs through the filter clear and colorless and the ppte. remaining assumes a pure gold-yellow color without any orange colored particles being present. The precipitate in the dish is now transferred to the paper, allowed to drain off, dried in the closet at 80-90 degrees C, the greater part of the ppte. is removed from the paper onto a watch glass, the paper replaced in the original funnel, and any particles remaining on the paper and in the dish is washed out by means of a jet of hot water into a weighed Pt or porcelain crucible, evaporated to dryness and the remainder of the ppte. on the watch glass put into the crucible. The whole ppte. is now dried at a 160 degrees C. and weighed. The calculation of the amount of KCl corresponding to the wt. of the ppte. is performed as follows: the weight P of the potassium chlorplatinate is multiplied by 0.3067 and gives at once the wt. of KCl. The wt. of the two chlorides, minus the wt. of the KCl equals the weight of the NaCl.

Analysis of Acid Radicals of Fuses

Chlorides, Chlorates, Perchlorates: in the mixture.

Chlorides: pipette 50 c. c. Sol. "B" into a 250 c. c. beaker, acidify with HNO_3 (2 c. c. (1-3) HNO_3) warm but do not boil, add 50 c. c. $AgNO_3$ of Approx. N/$_{10}$ sol. Stir vigorously to coagulate the ppte. and stand in a dark place for several hours (better stand over night).

Filter through a weighed Gooch, dry for an hour on the steam plate and weigh at AgCl. This gives chlorides alone.

Chlorates: pipette 50 c. c. Sol. "B" into a 250 c. c. beaker dilute to about 150 c. c. and treat with 5 to 10 c. c. of a 40% solution of formaldehyde, 2 c. c. of dilute HNO_3 (1-3), and aprox. 50 c. c. N/$_{10}$ sol. $AgNO_3$. The beaker containing the mixture is covered with a watch glass and heated on the steam bath for 3-4 hours, after which the ppte. AgCl may be at once filtered off, washed, dried and weighed. The period of heating on the steam bath may be shortened to 2 hours if the mixture is afterwards allowed to stand over night before filtering. Boiling the solution causes loss of some chlorine compound and makes the results low. This gives chlorides and chlorates.

Perchlorates: pipette 50 c. c. Sol. "B" into a 90 c. c. beaker, evaporate to about 10 c. c. transfer to a 25 c. c. Pt. crucible where evaporation is continued until the volume is about 5 c. c. Anhydrous Na_2CO_3 is then added to the solution in the crucible until the crucible is completely full of powder. The small amount of the solution is absorbed by the sodium carbonate, and is readily dried out by careful heating with a small flame or on a steam plate, after which the mass is heated over a burner until no evolution of gas bubbles is noted. The time of fusion is usually 15-20 minutes. The melt is then cooled, dissolved in dilute HNO_3 and the chloride determined by precipitation with $AgNO_3$. This gives chlorides, chlorates and perchlorates.

Determination of Acetic Acid in Sodium Acetate

Method: Introduce into a 500 c. c. long neck Kjeldahl flask a 2/3 gram sample of finely divided Sodium Acetate and wash all particles down sides of the flask with a little distilled water. Connect the flask with the condenser by means of a distilling trap, and then insert a dropping funnel in the flask so that the end is at the bottom. The distilling trap effectually prevents the carrying over mechanically of any phosphoric acid. Through the dropping funnel add 12-15 c. c. of the syrupy 85% phosphoric acid and 75 c. c. distilled water. Distil rapidly and collect the distillate in a 500 c. c. Erlenmeyer flask containing a little distilled water and an excess of standard normal alkali, being sure that the end of the condenser is below the surface of the alkali solution. Allow 15 c. c. of normal alkali for every gram of Sodium Acetate used, this will give a reasonable excess for the back tritation. The air inlet on the dropping funnel and outlet on collection flask should be protected with soda lime tubes.

Distil to a volume of 15 to 18 c. c. in flask at which point slight fumes are noticeable. Remove flame and allow the liquid to cool down before adding water. *Caution*: Be sure the stop cock in the dropping funnel is open when the liquid is cooling to prevent sucking back of the distillate into the distilling flask. When cool, add through the funnel 50 c. c. water and again distil to slight fumes. Cool and repeat with 50 c. c. more water.

When through distilling, disconnect condenser and rinse with distilled water. Remove collection flask and titrate the excess alkali in distillate with $N/_5$ HCl and phenolphthalein. Correct for chlorides determined on a separate sample and calculate as anhydrous Sodium Acetate.

Calculation:

$$\frac{\text{c. c. } N/_1 \text{ NaOH—c. c. acid in back titration} \times .082 \times 100 \quad \text{(calculated to } N/_1\text{)}}{\text{grams of sample}} = \text{per cent. anhydrous sodium acetate}$$

Determination of Chlorides in Commercial Sodium Acetate

Method: A 3-gram sample is weighed into a small beaker and dissolved in 100 c. c. distilled water, warming slightly. Filter to remove any insoluble matter and make exactly neutral with acetic acid. Add 5 c. c. of 5% potassium chromate solution and titrate to first trace of red with N/10 silver nitrate solution. Calculate as per cent. sodium chloride.

$$\frac{\text{c. c. N/10 } AgNO_3 \times .00585}{\text{Weight of sample}} = \%\ NaCl$$

To correct for chlorides in Sodium Acetate result

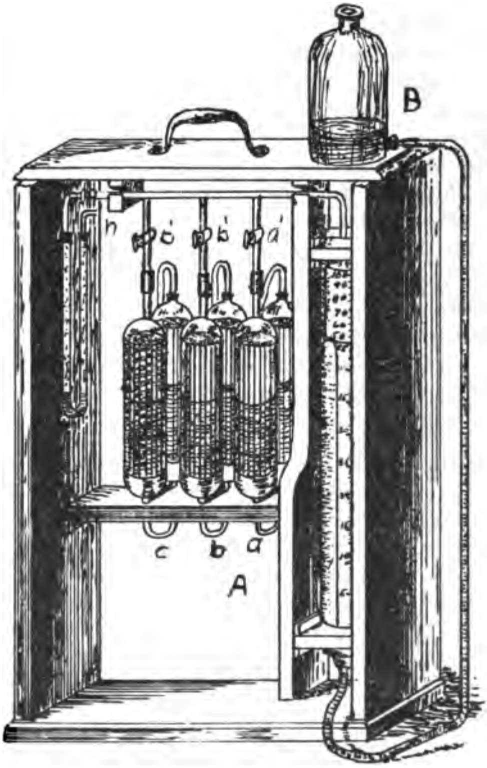
$\%NaCl : x = 58.5 : 82$

%Sodium Acetate found above — x = Correct per cent, anhydrous sodium acetate

Moisture: Moisture was determined by drying a 10-gram sample at 105° C. to constant weight.

Method of Determining Flue or Smoke Box Gases from Locomotives and Flues

SAMPLING



The gas is drawn from the smoke box through a tube extending from the front of the engine to the center of the area between the steam pipe and the exhaust pot. The tube is made of wrought iron, "T" shaped at the intake end, the "T" being perforated to such an extent that the total area of the perforation is not greater than the inside area of the sampling pipe.

The sample is collected by suction drawing the gas into a two liter glass bottle "C". The bottle is first completely filled with a saturated salt solution and connected by means of a siphon to a bottle of similar size. After the air has been exhausted from the tube a′, b′, and c′ connecting this tube with the pipettes a, b, and c respectively. The measuring burette is connected with a leveling bottle "B" by means of a flexible rubber tube.

Pipette b and c are connected at the rear to rubber bulbs which receive the excess air when pipettes are filled with gas and prevent the solution weakening by the absorption of oxygen from the air.

MANIPULATION

The capillary tube is first filled with a sample of the gas to be analyzed. This is then expelled to insure the absence of air. The three-way cock allows it to be done very easily. Draw into the measuring pipette a little over 100 c. c. of the gas by lowering the bottle "B" and opening cock h. Turn cock h so that gas can be expelled and bring bottle "B" to level of water in measuring tube at 0 c. c. The gas is thus measured at atmospheric pressure.

Close cock h, open cock a′ and run gas into pipette a by raising bottle "B". When the gas is in pipette a, lower bottle "B" and withdraw gas into measuring tube. Repeat twice and finally draw gas into measuring burette, close cock a′ and bring the liquid in the bottle to the level of the liquid in the tube. Take reading. Run gas into pipette and back again until reading is constant. The final reading gives the percentage of carbon dioxide in the flue gas.

Run gas into pipettes b and c in like manner and take readings. Subtract previous readings for carbon dioxide and oxygen and get percentage of oxygen and carbon monoxide respectively.

The nitrogen content is taken by difference. The residual gas is expelled and the apparatus is ready for another analysis.

Solutions: In pipettes a, b, c.

(*a*) Caustic Potash Solution: Dissolve 100 grams pure KOH in 300 c. c. water. Let it settle and use only the clear solution.

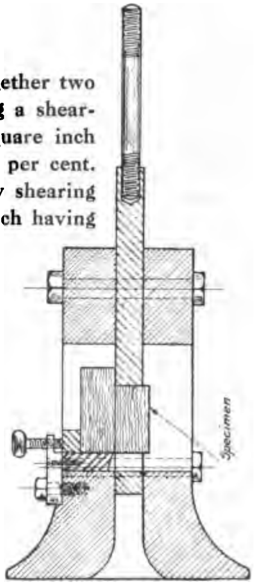
(*b*) Pyrogallic Acid Solution: The white re-sublimed acid should be used. 15 grams are dissolved in 150 c. c. KOH solution in *a*.

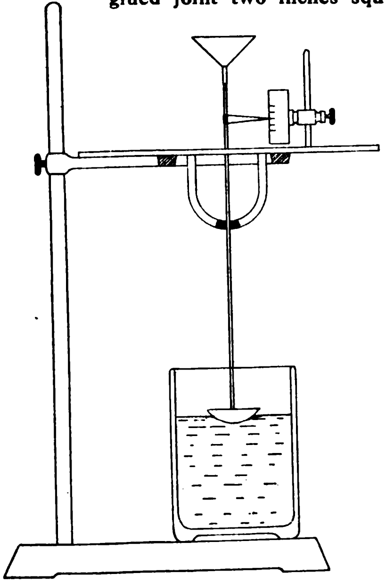
(*c*) Ammoniacial Cuprous Chloride Solution: 200 grams of NH_4Cl are dissolved in 750 c. c. water, in a bottle provided with a good rubber stopper, and 200 grams of cuprous chloride are added. When ready for use, mix with one-third of its volume of ammonia (Sp. Gr. .900).

Glue Tests

1. Strength Test.

The strength will be made by gluing together two pieces of yellow birch one inch thick, having a shearing strength of at least 2200 pounds per square inch and a moisture content of between 8 and 12 per cent. of the oven-dry weight, and testing them by shearing in a testing machine. (This will require birch having an air-dry weight of about 50 pounds or more per cubic foot.) The glue will be mixed in proportion of two parts of cold water to one part of glue and allowed to stand until the glue is soaked up. It will then be melted on a water bath and applied to the wood at a temperature of about 140° F. (60° C.). After gluing, the test blocks will be held in clamps for 24 hours, then remove from the clamps and allow to stand for six additional days. They will then be finished so that the blocks when ready to test will have a glued joint two inches square. When tested in this way no test block shall fail under a load of less than 2200 pounds per square inch. Four tests of glue shall be tested. If the shearing strength of the particular wood used is lower than 2200 pounds per square inch, it is evident that the results on the glued blocks will be lower.





2. Jelly Strength.

Twenty grams glue and 240 grams water. Solution made as under strength test. Let stand over night keeping cool in water bath or cool room, and test at 20° C. with jelly tester, recording the weigt necessary to depress the tester 0.125 inch.

he results obtained above are only comparative, since no two :esters will give results that check with each other. It is very tant that the beakers used with this apparatus are all of the size, since different size beakers give different results. For this ı it is desirable to use for this purpose drinking glasses of the or ordinary table use. Since the results are only comparative workers prefer to compare the sample with a standard by means ssing with the finger upon the jelly.

Odor.

he odor of the glue when in the hot solution must be sweet and remain sweet for 48 hours. Make this test on the samples or viscosity test.

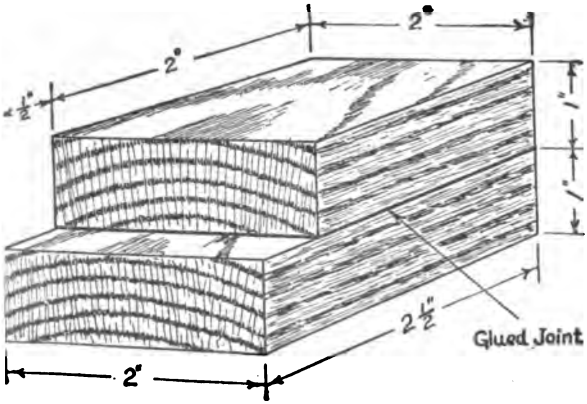
Ash.

sh 2 to 3 grams. Make a qualitative test for phosphates. If ıt in any quantity, determine as follows:

issolve ash in 85 c. c. water and 2 c. c. of HNO_3 (Sp. Gr. 1.42). 5 grams NH_4NO_3 and heat to 60° C. Add from a burette. 20 molybdic acid solution with stirring. Let stand 15 minutes. , wash well with cold water. Tear paper in 5 pieces, dissolve n 30 c. c. $N/_2$ NaOH. Dilute, titrate with $N/_2$ acid using lphthalein as indicator. 1 c. c. $N/_2$ alk equals .001545 gram P_2O_5.

Moisture.

ry about 2 grams of pulverized sample for 18 hours at 100° C. This test may be omitted unless specially desired.)



Total Soluble Chlorine:—Titrate 25 c. c. of the solution with standard silver nitrate, using potassium chromate as an indicator. Subtract the amount of chlorine equivalent to the free hydrochloric acid present, if any, and also the chlorine combined as ferric chloride, and calculate the remainder to zinc chloride, by using the factor, 1.92.

The amount of zinc chloride thus obtained should check with that found from Soluble Zinc.

Shellac Varnish

TOTAL SOLIDS

Weigh 2 grams by difference in a pint friction top lid. Dry 3 hours at 100° C. Cool and weigh. Loss is volatile thinners.

VOLATILE THINNERS

Distill 100 grams of the shellac varnish until incipient decomposition. Redistill the distillate noting the temperature and determine the specific gravity of the distillate. By this means it is usually easy to determine whether the varnish is made with wood alcohol, grain alcohol, or denatured alcohol. Dilute a portion with three times its volume of water; if benzin is present it will separate.

ANALYSIS OF SHELLAC

Weigh out sufficient shellac varnish to give .2 gram shellac. Evaporate until almost dry, add 10 c. c. glacial acetic acid and 10 c. c. chloroform. Be sure that the shellac is in complete solution. Add Hanus iodine solution and determine the iodine number in the usual manner. Run similar determinations on shellac varnishes of known composition made from pure shellac and alcohol and pure shellac and alcohol and pure rosin and alcohol and from mixtures of shellac, rosin and alcohol. Pure shellac should give an iodine number of not over 30, figured on the shellac present. Rosin will give an iodine number of about 230. Mixtures will give iodine numbers between the above values. When taking the iodine number of samples high in rosin the sample should be reduced to .1 gram to .15 gram.

If desired, the methods given by the American Society of Testing Materials may be used. These methods call for a Wijs' iodine solution, and give values of 18 for shellac and 228 for rosin, but it has been our experience that the Hanus method gives satisfactory results, and is much simpler since the Hanus solution is usually a stock solution.

MOISTURE IN BLEACHED SHELLAC

Dry 5 to 10 grams sample in a well ventilated oven for 6 hours at a temperature of 100° F. to 110° F. One or two electric light bulbs provide a convenient source of heat. Dry to constant weight. Dry shellac is very hygroscopic and should be weighed rapidly.

Bleached shellac may contain from traces up to 30% moisture.

Method of Examining Plush

The sample of plush to be examined for the tensile strength of the linen warp and cotton filling; for weight of plush, cotton and linen, per square yard; for number of cotton and linen threads per inch; for number of free ends of mohair per square inch, and average number of hairs per loop; for amount of free acid and potash per square yard; for thickness of plush and for permanency of dye or tendency to crock.

The weight of plush, cotton, linen, ash, and amount of free acid are all reported upon the basis of the square yard. For convenience of operation and calculation, it has been found best to use a factor size for obtaining these results:

1 square yard equals 1296 inches.
1 gram equals 28.35 ounces.

Weight of 1296 square inches, in grams, ÷ 28.35 equals weight of 1 square yard in ounces.

Or, weight of 45.714 square inches in grams (1296÷28.35) is the same figure as the weight of 1 square yard in ounces. A factor size would, therefore, be 45.714 square inches. It is, however, more convenient to use a sample one-fifth the size computed to 9.143 square inches. This piece of plush for examination is obtained by means of a square brass form, or template, measuring 3.024 inches along each edge and having an area of 9.143 square inches. This template consists of a plate fitted with dowel pins near each corner, which will exactly fit into a hole in a smaller plate, which serve to hold the plush in position while cutting. Both plates have their edges beveled on the back, to facilitate cutting the plush to the required size with shears.

In calculating the ounces per square yard, it is necessary to multiply the weight of the factor size by 5.

From the sample of plush received, cut three pieces measuring 9.143 square inches each. Weigh one piece and separate the linen threads of the warp from the cotton threads of the filling, weigh each separately, and report ounces of plush, ounces of linen, ounces of cotton per square yard, and the weight of the mohair by taking the sum of the weight of the cotton and linen threads from the weight of the plush. The second piece is burned in a porcelain or silica crucible, the ash weighed and calculated to ounces per square yard. The third piece is boiled for 5 minutes in 150 c. c. distilled water, the piece of plush removed from the beaker and thoroughly squeezed and the solution titrated with $N/_2$ potassium hydrate solution, using phenolphthalein as indicator. The number of c. c. taken equals the amount of potassium hydrate solution required to neutralize the free acid in the piece tested.

To determine the tensile strength of the cotton and linen thread, cut two pieces of plush, from 6 to 8 inches long, and 1⅛ to 1¼ inches wide. These should be cut longitudinally, one with the linen and the other with the cotton thread, bringing the piece down to exactly 1 inch in width by unraveling from both sides to make sure that there are no cut threads. The bringing down to size should not be done by shears alone, as it is almost impossible to avoid cutting one or more threads. These pieces are each in turn clamped in the jaws of the tensile test machine. The number of pounds required to break the sample is registered on the dial. The height of pile of the mohair is obtained by means of a micrometer gauge. The shade must compare favorably with a piece of standard plush. The crocking is made by rubbing a piece of plush back and forth several times on a piece of white paper. There should be no stain on the paper.

The number of linen threads and cotton threads per inch are counted by means of a magnifying glass or thread counter, with the brass plate having a ½ inch opening. The number of free ends of mohair is calculated by multiplying the number of linen threads by half the number of cotton threads per inch. This method of calculating is dependent upon the structure of plush made with a "W" weave. The number of hairs in each loop is counted by means of a dissecting microscope. Ten loops representing ten different mohair threads taken at random across the piece are dissected under the microscope and the number of hairs counted. The average number of hairs obtained by this method is taken as a measure of the fineness of the mohair.

Method of Analysis of Turpentine Substitute

The turpentine substitute is analyzed for color, specific gravity, residue and percentage of insoluble matter in sulphuric acid, exactly as given in "Method of Analyzing Turpentine." The percentage of distillation below 338 degrees Fahrenheit is not obtained.

In addition, the substitute is examined for flashing and evaporating qualities as follows:

FLASHING POINT

The flashing point is taken as described in "Methods of Determining Flashing and Burning Points of Combustible Liquids." The substitute should not flash at a temperature below 102 degrees Fahrenheit.

EVAPORATION

Place 2 c. c. of the sample on a large watch glass. On another large watch glass place 2 c. c. of a standard substitute. Let them stand side by side at room temperature. The sample examined should not require any longer to evaporate completely than the standard substitute.

DISTILLATION

100 c. c. of sample is distilled in the same manner as given in the "Method of Analysis of Turpentine," except the rate of distillation should be between 4 and 5 c. c. per minute. The boiling point is not determined, since the flash point takes care of low boiling constituents in a better manner. The distillation is carried out noting the temperatures at which the various fractions distill over, noting the temperatures for 5, 10, 20, 30, 40, 50, 60, 70, 80, 90 and 95%. This is sufficient for all ordinary work. In special cases 1000 c. c. of sample is distilled until 100 c. c. remain. This 100 c. c. is distilled in the above manner to determine the properties of the high boiling 10% fraction of the sample.

Method of Analysis of Turpentine

COLOR

The color of the turpentine is noted. It should be water white.

SPECIFIC GRAVITY

The specific gravity of the turpentine is taken by means of a Westphal's balance, first adjusting the temperature to 60 degrees Fahrenheit (15.6 degrees Centigrade). A description of this procedure is given in the "Method of Determining Specific Gravity of Oils and Other Liquids."

RESIDUE

Ten grams of the turpentine are weighed into a tared platinum evaporating dish and the turpentine is then evaporated over an ordinary water bath for four hours at a temperature not exceeding 212 degrees Fahrenheit. The dish is then cooled in a desiccator and weighed. The increase in weight is the residue. This weight is grams, divided by 10 and multiplied by 100, gives the percentage of residue left after evaporation.

PERCENTAGE OF INSOLUBLE IN SULPHURIC ACID

About 6 c. c. of the turpentine are placed in a 50 c. c. tube, graduated to tenths of a c. c. Allow it to stand 5 minutes and then note and record the exact volume of turpentine used, reading the volume to tenths of a c. c. For every c. c. of turpentine, 5 c. c. of concentrated sulphuric acid are now added to the contents of the tube e. g. if 6 c. c. of turpentine are taken, 30 c. c. of acid should be added.) The tube is held under the cold water tap and the acid is added slowly as there is danger of the whole mass blowing out of the tube if it becomes too hot. After all the acid has been added, allow the whole mass to become cool and then cork the tube with a rubber stopper, and mix by causing the material to flow from one end of the tube to the other five or six times, cooling with water during the operation, if necessary. Now set the tube vertical, and allow it to stand at the ordinary temperature of the room, not less nor more than half an hour. At the end of this time note, in tenths of a c. c., exactly the amount of clear liquid above the bass. This number of c. c. divided by the number of c. c. of the sample taken, times 100, gives the percentage of insoluble matter in sulphuric acid, as specified for this test. (Note: Formula for Acid:—1 part fuming H_2SO_4+2 parts conc. H_2SO_4.

Boiling point and Percentage of Distillate below 338 degrees Fahrenheit.

Measure 100 c. c. in a 100 c. c. graduate, and pour into the 200 c. c. distilling flask. Place a stopper holding a thermometer in the bulb in such a position that the bulb is opposite to the delivery of the distilling flask. Connect the distilling flask with a condenser tube and place the 100 c. c. graduated cylinder used in measuring the sample, without drying it, under the delivery end of the condenser tube to collect the distillate. With a low flame, bring the contents of the distillation flask slowly up to the boiling point and note the temperature at which the distillation begins. This temperature is recorded as the boiling point.

Now place a low flame, sufficient for maintaining distillation, under the distilling flask and surround the flask and flame with a shield to protect them from air currents. Continue the distillation thus, until the temperature has reached 338 degrees Fahrenheit. Then remove the flame and allow the last drops of distillate to drain from the condenser tube, and note the number of c. c. of distillate secured. Since 100 c. c. of the sample was employed, the number of c. c. of distillate secured is the percentage of distillate by volume, below 338 degrees Fahrenheit. The distillation, proceeding at a uniform rate, should require about 10 minutes from the time the first drops appear until the 338 degrees Fahrenheit temperature is reached.

Gasoline and Kerosene

GASOLINE

This method is abstracted from Technical Paper 166, Petroleum Technology 39, Bureau of Mines, Motor Gasoline Properties, Laboratory Method of Testing, and Practical Specifications by E. W. Dean.

The desirable properties of gasoline are:

(1) The gasoline should not contain too large a percentage of highly volatile products, which tend to cause large evaporation losses and excessive danger in handling and storage, but should have sufficient volatile constituents to permit starting an engine under reasonable conditions without preheating.

(2) The gasoline should not contain any considerable percentages of heavy or nonvolatile constituents, which after atomization into the engine cylinders can not be completely vaporized and burned.

(3) The gasoline should not contain a material which after combustion leaves a residue that collects in the motor.

(4) The gasoline should be free from substances that attack metal, either before or after combustion. Unremoved acid (used in refining) falls under this head.

(5) Neither the gasoline nor its products of combustion should have a strong or markedly disagreeable odor, because of this being objectionable to users of automobiles.

(6) The gasoline should be free from non-combustible material, such as water, sediment, acid, etc.

These stated requirements are simple in principle and are almost axiomatic. The chief problem is to fix limits, defined by actual tests, that will satisfy the desirable conditions.

Methods of Testing:

Color:—Examine sample in clear glass bottles.

Odor:—Gasoline should be free from rank and disagreeable odors as these cause discomfort to users.

Water, Sediments and Other Foreign Matter—Gasoline should be free from water, sediments and other foreign matter. Water is seldom present in gasoline and is easy to detect as the two liquids are mutually insoluble.

Acidity:—Acidity may be determined by shaking sample with distilled water and testing with litmus paper. Most gasolines are free from acidity.

Calorific Power or Heating Value—No data is at hand to show effects of calorific power on the usefulness of gasoline. Since only a small fraction of the calorific power is finally developed as mechanical energy in the engine, this determination is usually unnecessary.

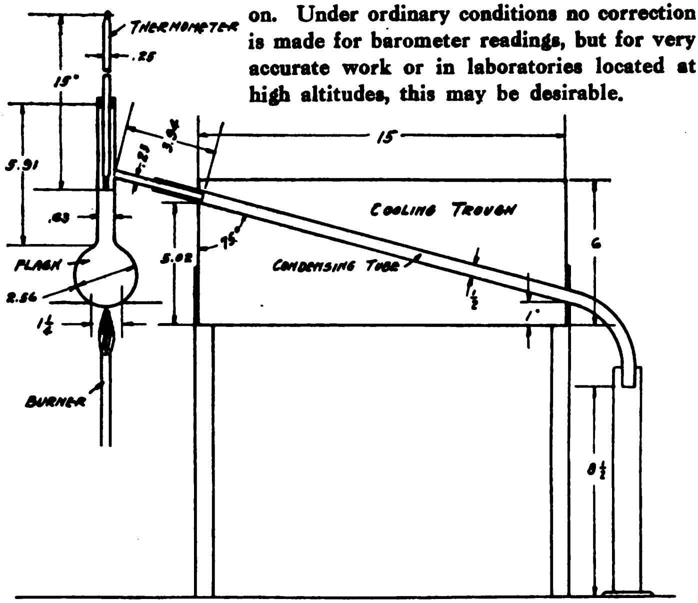
Aromatic Hydrocarbons—Present indications are that aromatic hydrocarbons are just as good motor fuel as non-aromatics. Aromatic hydrocarbons of the same range in boiling point as non-aromatic hydrocarbons have much higher specific gravity, approximately .87 to .88 for the aromatics as compared with .70 to .76 for the non-aromatics.

Unsaturated Hydrocarbons—This Bulletin (Technical Paper 166) states that the effect of unsaturated hydrocarbons on the properties of gasoline has not been worked out completely and that moderate percentages of unsaturated hydrocarbons can be used with entire satisfaction, but whether or not unlimited proportions can be used is not known. It further states that the measurement of the percentage absorbed by ordinary cold sulphuric acid seems to be the most desirable method of determining the unsaturated hydrocarbons. Later experiments since the publication of this Bulletin indicates that unsaturated hydrocarbons should not be present in amounts over 7%.

Specific Gravity—This determination is of very slight significance in determining the properties of gasoline, but it is a desirable test. The ordinary hydrometer or Westphal Balance may be used in determining specific gravity.

Volatility—This is the most important single test on gasoline. The determination of volatility may be made according to the method of analysis as specified for turpentine substitute, Method No. 38.

In addition it is sometimes desirable to record the boiling point and dry point. The boiling point is variously taken as the point where the first drop appears at the end of the condenser, the fifth drop at the end of the condenser, or when 5 c. c. has collected in the receiving flask. If the boiling point is taken, the method should be recorded. The dry point is taken at the point where the last drop is vaporized. The apparatus which is recommended is shown in the sketch. The thermometer used in this determination should be an accurate nitrogen filled "total immersion" instrument with a short bulb and with the mark for 35° C. or 95° F. at a distance between 100 and 120 mm. from the top of the bulb. These requirements insure that almost always the lowest temperatures registered may be read above the cork of the distillation flask. The "stem correction" should not be applied and it is understood that results of distillation are expressed in terms of thermometer readings but not of actual temperatures. It is also desirable to record the barometer readings to avoid any disputes later on. Under ordinary conditions no correction is made for barometer readings, but for very accurate work or in laboratories located at high altitudes, this may be desirable.



APPARATUS FOR THE DISTILLATION OF GASOLINE.

The gasolines from different sections of the country vary somewhat in their properties and the character of gasoline changes from year to year. Below is given the analysis of a sample of commercial automobile gasoline obtained at a street filling station in Dayton, Ohio, February, 1919.

DISTILLATION

1st drop	140° F.
5%	174° F.
10%	187° F.
20%	205° F.
30%	221° F.
40%	239° F.
50%	248° F.
60%	264° F.
70%	282° F.
80%	304° F.
90%	340° F.
95%	366° F.
Specific gravity at 60° F.	.748

KEROSENE

Kerosene is analyzed in the same manner as gasoline except for the changes made necessary by the higher boiling tmperatures of kerosene. In addition, the flash point on kerosene should be determined and, if the kerosene is to be used for illuminating purposes in a lamp, it is desirable to make an actual practical burning test in the lamp, since slight impurities resulting from imperfect refining may seriously interfere with the use of kerosene in lamps. This burning test should be continued for a long period of time, frequently a week or more depending upon conditions.

Sand Analysis

1. Loss on Washing:—Weigh up 200 grams of the sand as it comes into a liter beaker on the rough balance. Fill the beaker with water and stir rapidly to get the removable matter suspended in the water, allow the sand to settle out (which it does very quickly) and decant off the liquid taking care not to pour off any sand. Repeat the washing until the water above the sand remains clear after vigorous stirring. By this time all clay and removable matter will be washed out, drain off all the water from the sand that is possible and set the beaker on the hot plate, on a piece of asbestos until dry. Cool and weigh the beaker and contents. Calculate the % loss.

2. Loss on Ignition:—Weigh .5 gram of the powdered sample into a platinum crucible and ignite strongly in the Electric muffle for 2 hours. Cool and weigh, repeat for 1 hour, to see whether a constant weight has been obtained. The loss in wt. $\times 2 \times 100 = \%$ Loss on Ignition.

3. Moisture:—Weigh .5 gram of the powdered sample on a watch glass with care and put it on the hot plate for 2 hours. Cool and weigh. Loss $\times 2 \times 100 = \%$ moisture.

4. Caking Test:—Fill a 4 inch watch crystal with the coarse sand and with a small jet of water moisten it until the water begins to siep through to the watch crystal, put the watch crystal on the steam plate and let it dry for several hours. If any clay is present, the sand will cake. Care must be taken so that no more water than is necessary to wet the sand through is added, or else the clay will be washed through and form a crust on the crystal.

5. Water Soluble:—Weigh 10 grams onto a counterpoised filter paper, place in a funnel and wash with distilled water, dry and reweigh. Note loss in weight. Test the filtrate for Cl, with $AgNO_3$: and SO_3 with $BaCl_2$.

6. SiO_2:—After the loss on ignition is obtained in 2, treat contents of crucible with 3 drops H_2SO_4 and 2 c. c. HF and stand the crucible on the steam plate until all HF has evaporated. Heat over free flame to drive off H_2SO_4. Weigh and get % loss, treat residue with 1 gram Na_2CO_3 and fuse. Take up fusion in HCl (dilute). Evaporate to dryness: to dehydrate silica. Take up in dilute HCl and filter off SiO_2, ignite and weigh, add to loss obtained by HF for total SiO_2.

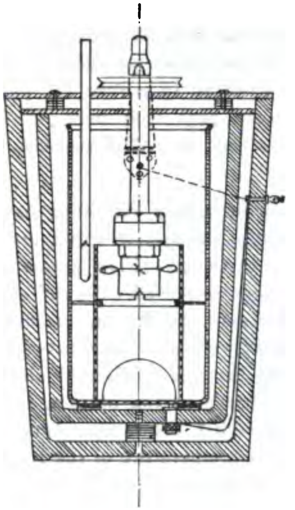
Sponges

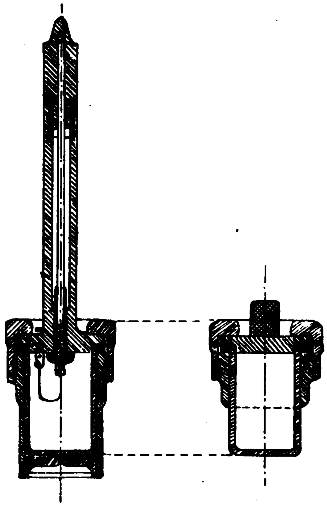
Weigh the sample of sponge as it comes in, as accurately as possible. Place it in a large evaporating dish and cover with water. Allow it to remain thus until it is thoroughly soaked and washed and soft. Then wash out all that is possible in one washing. Pour this washing into a weighed liter beaker and evaporate to dryness. Weigh the residue and set this aside for the present. Continue washing the sponge under the tap, until it is free from all dirt. Press out all the water that is possible, and hang over the hot plate to dry. Weigh and get the percentage of loss.

Digest the residue in 100 c. c. of water for an hour or two, filter and examine the filtrate for sugar. If Fehling's Solution fails to give any test for sugar, treat in the following manner: Add enough sub-acetate of lead to ppte. all the Albumen, filter and add enough potassium sulphate to ppte. the excess of lead, again filter, and with the clear filtrate proceed to test for sugar with Fehling's solution.

The sugar is in the form of cheap molasses or burnt sugar, it is added by the sponge collectors to give weight to the sponges, as the sponges are sold by weight. In addition to sugar as natural adulteration, is found: gurry, salt, clay, weeds, grass, shells and water, these substances being found in sea water. If the residue from the washing is dark or black, it is a good indication of intentional adulteration, as sugar is nearly always found. In some samples I have found as high as 70% removable matter by washing, 30% is the limit specified in good specifications.

Coal Analysis





Moisture:—Weigh 1 gram of finely ground sample into a weighed platinum crucible (the wt. of the lid included) and heat for 1 hour in the Toulene bath, at a temperature of 100-106 degrees C. Cool and weigh at once. W_1-W_2=loss in weight or moisture, divided by wt. of the sample × 100=% of moisture.

Volatile:—Weigh 1 gram sample into a weighed platinum crucible and cover with the lid. Heat for 7 minutes over a Bunsen flame. Cool and weigh. Loss in weight—moisture×100=% Volatile.

Carbon:—C=100—(M plus V plus A)

Ash:—The sample used for moisture is ignited strongly for 2 hours. Cool and weighed. The wt. of the ash divided by 1×100 =% Ash.

B. T. U.

The Parr Calorimeter has the advantage of operating without an oxygen gas supply. Its manipulation is simple and the results are accurate to within about 0.5%, making the results comparable with those obtained by the more expensive bomb calorimeters. The apparatus is a copper vessel of a little over 2 liters capacity, insulated by two outer vessels of fibre so as to provide burther insulation. The cover is double to correspond with an air space between the two parts. The sample is burned in the small cartridge shown in the center drawing. This cartridge rests on a pivot below and extends through the covers above and has a small removable pulley at the end. Turbine wings fastened to spring clips are placed on the cartridge and a short cylinder open at both ends is provided for directing the current set up by rotation of the vanes attached to the cartridge. The stem of the cartridge is arranged to permit the passage of a piece of about No. 34 American gage iron wire, 4" in length. The two ends of the cartridge screw on. The process is then as follows:

One-half gram of coal ground to pass through a 100-mesh sieve and dried in the oven as usual at 105 to 110° C. is mixed with 1 gram of potassium chlorate and placed in the cartridge. Five grams of sodium peroxide are added, and the cartridge is closed with the cap. It is then thoroughly shaken to mix the contents and the cap removed and replaced with the stem carrying the iron wire which projects into the sample. The peroxide should all pass through a 25-mesh sieve. After tapping the cartridge to settle the charge in the bottom, it is placed in the can and two liters of water added, the temperature of the water being 3 to 4 degrees lower than the temperature of the room. The pulley is attached to a motor and is rotated at about 100 revolutions per minute. When constant temperature is reached, the reading of the thermometer is taken and the charge is ignited by passing a current through the iron wire. After about 3 minutes, the thermometer should be watched constantly until the maximum temperature is reached, which ordinarily requires about 5 minutes. The maximum reading of the thermometer is taken. Duplicate results should check within .02° C.

Sulphur—The fused mass is now removed from the bomb to a porcelain dish, washing well. Bromine water is now added, and the solution made acid with HCl. The iron is precipitated by the addition of ammonia, boiled and filtered. Make the filtrate slightly acid with HCl and precipitate the Sulphur as $BaSo_4$ with a 10% solution of $BaCl_2$.

Weight $BaSO_4 \times .1373 \times 2 \times 100 = \%$ Sulphur.

The calculations for B. T. U. are made in accordance with the directions furnished with the instrument. A sample of these calculations is as follows:

Correction for Apparatus = .345° C.
Correction for Sulphur = .010 × % Sulphur.
Correction for Ash = .005 × % Ash.
Degrees temperature rise — correction, × 3100 = B. T. U.

$$\frac{\text{B. T. U.} - (4050 \text{ wt. of Sulphur})}{1 - (\text{wt. A plus S plus M})} = \text{actual B. T. U.}$$

M = .60 and .62 Average = .61
V = 23.98 and 23.96 Average = 23.97
C = 69.92 and 69.96 Average = 69.94
Ash = 5.50 and 5.46 Average = 5.48 × .005 = .0274
S = 1.78 and 1.76 Average = 1.77 × .010 = .0177

.0451

Rise = 4.98 and 4.97 Average = 4.975

$$4.975 - .390\ (.0177 + .345 + .0274) = 14{,}213 \text{ B. T. U.}$$

$$\frac{\text{B. T. U.} - (4050 \times \text{weight of S})}{1 - (\text{wt. A plus S plus M})} =$$

$$\frac{14{,}213 - (4050 \times .0177)}{1 - (.0177 + .0548 + .0061)} =$$

$$\frac{14{,}213 - 72}{1 - .0786} = \frac{14{,}141}{.9214} = 15{,}350 = \text{actual B. T. U.}$$

Analysis of Boiler Scale

Weigh 1 gram accurately into a platinum crucible with 4-6 grams of a mixture of Na_2CO_3 and K_2CO_3. Mix the contents well with a pt. rod. Heat gently for 10 to 15 minutes and finally over a Meaker burner for 1 hour. Remove the crucible and rotate it in such a way as to make the melt cool in a uniform layer on the sides of the crucible as well as on the bottom. Immerse the crucible $^2/_3$ the way into cold water while quite hot. This sudden cooling loosens the cake and it can be taken out. Put the cake into a casserole containing 150-200 c. c. H_2O, crush the lumps with a large stirring rod, and allow the solution to thoroughly disintegrate. Make slightly acid with HCl and evaporate on the steam plate to dryness; take up residue in water, add 20 c. c. HCl, evaporate to dryness, take up the residue in 100 c. c. HCl (1:4) and boil 10 minutes. Filter off SiO_2, wash with 5% HCl, hot, until all yellow stains disappear and finally with hot water. Bring the filtrate to a boil and make slightly alkaline with ammonium, boil a few minutes and pour through a 11 c. m. filter collecting the Fe and Al, wash well with ammonia wash, and with hot water. Collect the filtrate in a 200 c. c. flask and call it Solution "A".

If the sample contains P, redissolve the ppte. in dilute HCl (1:4). Cautiously add Na_2CO_3 Sol. drop by drop from a pipette until a permanent muddy color appears. Then 1 drop HCl to clear away the cloudy appearance. Add 20 c. c. ammonium acetate and boil 5 minutes and allow the ppte. to settle, filter off, put into pt. crucible, ignite and weigh. Dissolve contents of the crucible in strong HCl, dilute and ppte., filter and wash and set aside or reject filtrate. To the ppte. on the paper, pour 20 c. c. of a hot solution of (1:4) H_2SO_4 very slowly around the edges of the paper, drop by drop washing into a small beaker. Pass this solution through the reductor, and titrate with $KMnO_4$, compute % Fe. Al plus Fe—Fe=Al. This procedure separates the Fe and Al from the Phos. The Phos. must be calculated on a separate sample by the Molybd. method, or one of the standard methods.

SOLUTION "A"

CaO—(CaC_2O_4 as ppted.) Take 100 c. c. from flask into 350 c. c. beaker, dilute with 100 c. c. water and bring to boil and add 20 c. c. of 5% solution of boiling $(NH_4)_2C_2O_4$, continue to boil for a few minutes, when the CaC_2O_4 will separate out as fine crystals. Allow this to stand for 5-6 hours (better over night). Filter through a close filter, wash with ammonium oxalate wash water in the beaker several times, finally transfer to the paper and wash with hot water. Transfer the ppte. and paper to the pt. crucible and ignite carefully, and finally in the muffle to a constant wt. The Ca is in the form of the oxide CaO and is to be reported as such.

CO_2—On separate sample (.5 gram). Determine by absorption in KOH bulbs.

Magnesium—as $Mg_2P_2O_7$—To filtrate from the CaO, add 90 c. c. strong ammonium and 10 c. c. microcosmic salt solution and a few crystals of microcosmic salt. Stir well and allow this ppte. to settle out over night. Decant through a weighed Gooch, transfer ppte., wash with NH_4NO_3 wash water, dry and ignite carefully and weigh. Wt. times .36207=wt. MgO.

Sulphate: as $BaSO_4$ (Total and Free S—Treadwell Hall page 505). The filtrate from the magnesium is boiled to remove all excess of ammonia, made acid with HCl, 20 c. c. 10% $BaCl_2$ added, boiled for ½ hour and allowed to settle, stand ¾ hours. Filter and collect on a very fine paper washed with hot water, transferred to a pt. crucible and ignited wet, cool and weigh. Calculate SO_3.

The above analysis holds good for Silicates, Slag, and Carbonates. For the less common constituents, refer to Gov. Bulletin 422.

U. S. Geological Survey.

Determination of Borax and Boric Acid

* Sutton's Vol. Analysis page 93.

Dissolve 1 gram of sample in water, add methyl orange and dilute H_2SO_4 until pink color just appears. Boil to expel CO_2, cool and add standard NaOH until pink color changes to a pure yellow tinge. At this stage all the Boric acid exists in a free state. Add glycerine in such proportions that the total solution after titration will contain at least 30% of it. Then add a little phenolphthalein and lastly titrate with standard NaOH until a permanent pink is obtained. Now glycerine is added and if pink color disappears, add more NaOH. Continue this process until glycerine has no effect on the solution.

The Boric acid is calculated from the number of c. c. NaOH:

1 c. c. normal NaOH=.0620 gram H_3BO_3
=.505 gram $Na_2B_4O_7$
=.0955 gram $Na_2B_4O_7$ 10 H_2O.

Copper in Pigments

Dissolve .5 to 1 gram in 5 c. c. each of HCl, HNO_3, and 1:1 $H_2 SO_4$. Evaporate to white fumes, cool, add 20 c. c. H_2O, boil till Copper is in solution. Filter if lead is present. Add 40 c. c. H_2O and 4 square inches of sheet aluminum (free from copper). Add 5 c. c. H_2SO_4 and boil 15 minutes. Wash the precipitate Copper by decantation and dissolve in 5 c. c. HNO_3 (1.2 Sp. Gr.). Dilute. Pour off $Cu(NO_3)_2$ solution, boil to expel NO_2 fumes, make just alkaline with NH_4OH and add 3 c. c. C. P. acetic acid. Heat if necessary until copper is in solution. Cool to room temperature. Add 30 c. c. 10% KI and titrate with $N/_{10}$ Sodium Thiosulphate. Standardize against pure copper.

1 c. c. of $N/_{10}$ Sodium Thiosulphate = .00636 Cu.

Zinc Oxide in Pigments

ZINC OXIDE IN PIGMENTS

The zinc oxide is determined by the ferrocyanide method after removal of copper, iron and other interfering materials. Calcium barium, aluminum, lead and silica do not interfere.

Solution Required:

Potassium Ferrocyanide—Dissolve 100 grams of potassium ferrocyanide in water and put in a clean 5-pint acid bottle and fill the bottle with distilled water. Mix thoroughly, standardize before using.

Ammonium Chloride—Use a saturated solution.

Uranium Acetate or Uranium Nitrate—Use a 5% solution in water containing a few drops of hydrochloric acid.

Method of Standardization:

Ignite some C. P. zinc oxide and cool in a desiccator. Weigh ½ gram sample into a beaker and dissolve in 10 c. c. hydrochloric acid and 100 c. c. water. Neutralize with ammonium hydroxide. Make just acid with hydrochloric acid and add 3 c. c. more. Add 10 c. c. saturated solution of ammonium chloride. Heat nearly to boiling and titrate with potassium ferrocyanide, using uranium acetate or nitrate as outside indicator.

Method of Analysis:

The pigment is obtained in hydrochloric acid solution and freed from iron, copper or other interfering materials by the usual methods and is then titrated exactly as in the method of standardizing potassium ferrocyanide against zinc oxide.

Methods of Sampling and Analysis

1. SAMPLING DELIVERIES

(*a*) *Cake Soap.*—One cake shall be taken at random from each 1000 cakes to constitute the test sample, except when a sample so drawn shall weigh less than 1 pound and be less than three cakes, in which case additional cakes, to make a sample of not less than 1 pound and not less than three cakes shall be taken. The sample cakes shall be at once placed in an air-tight container, properly labeled, and sent to the laboratory for test.

(*b*) *Liquid Soap.*—After thorough mixing of the contents, 1 quart shall be drawn from one container taken at random from each delivery of 100 gallons or less of the material. If a larger volume of the material is delivered at one time, 1 quart shall be taken to constitute a sample for each 100 gallons. Each sample shall be at once placed in a quart glass bottle which has previously been thoroughly cleaned and dried, securely stoppered with a new cork, properly labeled, and sent to the laboratory for test. If desired the entire contents of one small container selected at random from each delivery may be sent as the sample.

(*c*) *Chip Soap.*—Not less than 1 pound taken at random from each 1000 pounds or less of any shipment shall constitute a sample. The sample shall be at once placed in a clean, dry, air-tight container, which must be completely filled with the sample, properly labeled, and sent to the laboratory for test.

2. PREPARATION OF LABORATORY SAMPLE

(*a*) *Cake Soap.*—Weigh each cake, set aside in an air-tight container one, or two cakes of about medium weight, with record of weights as received, for making check analysis if necessary. Select another cake of about medium weight, cut in two in a diagonal direction, and cut from these fresh surfaces, in thin layers, a sufficient quantity for all determinations, taking care to cut entirely across so as to get a fair proportion of the outer and inner parts. Preserve this sample in a tightly stoppered bottle and weigh the different portions required as soon as possible. Instead of preparing the sample by cutting, one or more cakes may be run three times, rapidly, through a meat chopper.

(*b*) *Liquid Soap.*—No preparation of the sample is necessary unless it is received during very cold weather, when it should be allowed to stand at least one hour after it has warmed up to room temperature (20° to 30° C.) before noting whether the material is clear, free from objectionable odor, and whether it forms a satisfactory lather.

(*c*) *Chip Soaps.*—Rapidly mix the sample and weigh out all the portions for analysis, preserving the remainder in an air-tight container.

3. METHODS OF ANALYSIS

The following methods are to be used in examining soap purchased under any of the specifications recommended in this circular:

(*a*) *Matter Volatile at 105° C.*—Place 2 grams of the sample in a weighed porcelain dish 3 inches in diameter, spreading the sample as evenly as possible over the bottom of the dish; dry for one hour at 60° C., add 50 c. c. of absolute alcohol, evaporate, dry for one hour at 60° C., and then for two hours in an oven at 105° C., cool in a desiccator, and weigh. Calculate percentage loss.

✓ (*b*) *Free Alkali or Acid* (*Method for All Except Liquid Soaps*).—Dissolve 5 grams of the sample in about 200 c. c. of freshly boiled, neutral, 95 per cent. alcohol, heating to complete the solution. Filter through a weighed Gooch crucible (if the sample is known to contain less than the specified amount of matter insoluble in water, a filter paper may be used), wash with hot, freshly boiled neutral alcohol. Unite filtrate and washings, cool, add phenolphthalein, and titrate with standard acid or alkali. Calculate the percentage of free alkali as NaOH or free acid as oleic acid.

(*c*) *Free Alkali or Acid* (*Method for Liquid Soaps*).—Dissolve 10 grams of the sample in about 200 c. c. of freshly boiled, neutral absolute alcohol. Filter and wash the precipitate with freshly boiled, neutral alcohol. Unite the filtrate and washings, add phenolphthalein, and titrate with standard acid or alkali. Calculate the percentage of free alkali as KOH or free acid as oleic acid.

✓ (*d*) *Alkali as Alkaline Salts.*—Extract with hot water the matter insoluble in alcohol which is separated during the determination of free alkali or acid, add methyl orange to the water solution, and titrate with standard acid (HCl). Calculate the percentage of alkaline salts as Na_2CO_3 (or as K_2CO_3 in the case of liquid soaps). (It is understood that the "alkali as alkaline salts" as thus determined includes all alkali in the form of carbonate, borate, and silicate.)

(*e*) *Silicate.*—Add an excess of hydrochloric acid to the solution remaining after titration of klaaline salts, evaporate to dryness, take up with HCl, and determine silica in the regular way. Calculate the equivalent Na_2CO_3 assuming the silica to be in the form $Na_2Si_4O_9$.

(*f*) *Sulphate.*—Determine the sulphate in the filtrate from the silica by precipitation with barium chloride and weighing as barium sulphate in the ordinary manner; calculate as sodium sulphate (Na_2SO_4), except for liquid soaps, which are calculated at K_2SO_4.

(*g*) *Matter Insoluble in Water.*—Dry the Gooch crucible used in the determination of alkali as alkaline salts for two hours in an oven heated to 105° C., weigh, and calculate the percentage of total insoluble matter.

(*h*) *Unsaponified Saponifiable Matter.*—Dissolve 5 grams of soap in about 100 c. c. of 50 per cent. alcohol; if any free fatty acids are present, add just enough standard alkali to neutralize them and wash into a separate funnel with 50 per cent. alcohol. Extract with 100 c. c. ether. Wash the ether with water, evaporate, and weigh unsaponified matter. Add 25 c. c. alcoholic potash or soda, boil under a reflux condenser for one hour, evaporate to dryness, dissolve in water, transfer with ether and water to a separatory funnel, wash the ether free from alkali, transfer to a weighed dish, evaporate the ether, dry, and weigh. This weight gives the unsaponifiable matter, which is subtracted from the unsaponified matter. To the remainder add the weight of free fatty acid previously determined; the sum is the total unsaponified saponifiable matter.

(*i*) *Preparation of Total Acids* (*Method for Milled-Toilet, White-Floating, or Salt-Water Soaps*).—Dissolve about 50 grams of the soap in 300 c. c. of hot water, transfer to a separatory funnel, add 150 c. c. of approximately 2 N H_2SO_4, cool somewhat, add 120 c. c. of ether, shake, draw off the acid layer, and wash the ether layer free from acid with a strong salt (NaCl) solution. Then draw off the aqueous layer as completely as possible, transfer the ether layer to a flask (it is not necessary to transfer quantitatively), add 20 to 30 grams of anhydrous sodium sulphate (Na_2SO_4), stopper the flask, shake, and let stand at a temperature below 25° C. until the ethereal liquid becomes perfectly clear, showing that all water has been taken up by the sodium sulphate. Filter through a dry paper into another Erlenmeyer flask, and completely evaporate off the ether by passing through the flask a current of dry air and heating the flask to a temperature not above 50° C.

(*j*) *Preparation of Fatty and Rosin Acids* (*Method for Ordinary or Special Grade Laundry or Chip Soaps*).—Dissolve about 50 grams of the soap in 500 c. c. of hot water, add 100 c. c. of 30 per cent. sulphuric acid, heat until the fatty matter collects in a clear layer, draw off the acid layer, and wash the fatty matter free from sulphuric acid with hot water. Decant the fatty matter into a dry beaker, filter, using a hot-water funnel, or placing both funnel and receiving beaker in a water-jacketed oven, and dry for 20 minutes at a temperature of boiling water.

(*k*) *Titer Test.*—Cool the fatty acids, prepared as described, to about 50° C. and transfer about 25 c. c. to a tube of clear glass about 1 m. m. thick, 25 m. m. in diameter, and 100 m. m. long. Place the tube in a salt-mouth bottle, about 70 m. m. in diameter and 150 m. m. high (a 16-ounce salt-mouth bottle), fitted with a cork which is perforated so as to hold the tube rigidly when in position. Suspend a thermometer graduated to 0.1° C. so that it can be used as a stirrer and stir the mass slowly until the mercury remains stationary for 30 seconds. Then allow the thermometer to hang quietly, with the bulb in the center of the mass, and observe the rise of the mercury. The highest point to which it rises is recorded as the "titer."

(*l*) *Acid Number of Fatty Acids.*—Place from 2 to 5 grams of the fatty acids, prepared as described, in a 200 c. c. Erlenmeyer flask, add 50 c. c. of neutral alcohol free from carbon dioxide, warm to dissolve the fatty matter, add phenolphthalein, titrate with standard alcoholic alkali, and calculate the acid number (expressed as milligrams KOH per 1 gram total fatty matter).

(*m*) *Total Alkali* (*Method for all Except Liquid Soaps*).—Dissolve 5 grams of the soap in 100 to 150 c. c. of hot water, add 40 c. c. $N/_2$ H_2SO_4, heat on a water bath until the fatty acids have collected in a clear layer, cool by placing the beaker containing the mixture in cold water, break the cake of fatty acids, and filter the aqueous liquid, leaving as much of the fatty acids as possible in the beaker; thoroughly was the beaker, fatty acids, and filter paper with water. Unite the filtrate and washings, add methyl orange, and titrate the excess acid with $N/_5$ NaOH. Calculate the total alkali as Na_2O.

(*n*) *Total Alkali* (*Method for Liquid Soaps*).—Warm together all of the acid aqueous liquids from the determination of total soap to drive off dissolved ether, add methyl orange and titrate the excess acid with $N/_5$ NaOH. Calculate the alkali as per cent, K_2O in original sample.

(*o*) *Chloride.*—Add neutral potassium chromate (K_2CrO_4) solution to the neutral solution from the titration of total alkali, titrate with $N/_{10}$ $AgNO_3$. Calculate the chloride as sodium chloride (NaCl) or as KCl in the case of liquid soaps.

(*p*) *Rosin.*—A qualitative test for rosin may be made as follows: After decomposing a solution of the soap and separating the fatty acids a small quantity of the latter is heated with acetic anhydride. When cool a few drops are placed on a spot plate and when a drop of H_2SO_4 (Sp. Gr. = 1.53) is added to this a figurative violet color indicates the presence of rosin. For a quantitative determination proceed as follows: Dissolve 5 grams of the sample in 100 to 200 c. c. of hot water, add 40 c. c. $N/_2$ H_2SO_4, heat until the fatty acids, etc., collect in a clear layer, cool in ice water, remove the layer of fatty acids, etc. Wash the fatty acids with ice water, transfer to a 200 c. c. Erlenmeyer flask, dry for one hour at 105° C., cool, dissolve in 20 c. c. of absolute alcohol, add 10 c. c. of a solution of one volume of strong sulphuric acid and four volumes of absolute alcohol, and boil for 4 minutes under a reflux condenser. Add to the liquid about five times its volume of 7 to 10 per cent. solution of NaCl and extract with ether. Shake out the aqueous portion two or three times with ether. Unite the ether solutions and wash with the sodium chloride solution until the washings are neutral. Titrate the rosin acids with standard alcoholic sodium hydroxide solution (1 c. c. normal alkali $=$ 0.346 g. rosin). Calculate percentage of rosin.

(*q*) *Total Soap* (*Method for Liquid Soaps*).—Dissolve 10 grams of the soap in 100 c. c. water, add 40 c. c. $N/_2$ H_2SO_4, heat until a clear layer of fatty acids separates, transfer to a separatory funnel, draw off the acid layer, and shake the acid aqueous liquid with two 20-c. c. portions of ether. Dissolve the fatty acids in the ether used for washing the aquaeous liquid, and shake with 10 c. c. portions of water until they are no longer acid to methyl orange. Unite the water portions used for washing and shake with 20 c. c. ether, wash this ether free from mineral acids, unite the ether solutions in a weighed 400 c. c. beaker, evaporate most of the ether at a temperature below 50° C., dissolve the residue in neutral alcohol free from carbon dioxide, add phenolphthalein and titrate to exact neutrality with standard alcoholic potash. Evaporate the alcohol, dry to constant weight at 105° C., and weigh the soap.

Instead of using standard alcoholic potash for this determination, standard alcoholic soda may be used; but in this case the weight of Na_2O added should be calculated from the volume of standard NaOH used in titration and one-half of this weight added to the determined weight of soap before calculating the percentage.

If unsaponified or unsaponifiable matter is suspected, the determination may be corrected by dissolving the dried soap in 50 per cent. alcohol, shaking out with ether, evaporating the ether and weighing the unsaponified and unsaponifiable matter. This weight is to be deducted from the weight of anhydrous soap before calculating percentages.

(*r*) *Sugar—Qualitative Test.*—Add a decided excess of hydrochloric acid to a solution of the soap, heat on steam bath for fifteen minutes, cool, filter from fatty acids, and test a portion of the filtrate for sugar by boiling for two minutes with an equal volume of Fehling solution. The formation of red cuprous oxide indicates sugar.

Methods of Determining Free Caustic, Carbonated, Combined and Total Alkali in Soaps

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FREE CAUSTIC ALKALI

Dissolve 5 grams of soap cut in fine shavings in 100 c. c. of an alcoholic solution of stearic acid (8 grams stearic acid per liter of 95% alcohol). Heat to boiling until soap is dissolved. Filter off the insoluble carbonates, etc., wash well with neutral 95% alcohol free from CO_2, and titrate the excess of stearic acid with $N/_2$ alkali, using phenolphthalein as indicator. Run a blank and calculate the percentage of alkali as NaOH or KOH or free acid as oleic acid.

1 c. c. $N/_2$ alkali = .020 gm NaOH = .0155 gm Na_2O = 0.1412 gm oleic acid = .02805 gm KOH = .02355 K_2O.

CARBONATED ALKALI

The residue from above is dissolved on the filter with hot water, using about 100 c. c. and titrate with $N/_2$ acid, using methyl orange as indicator after cooling; or an excess of $N/_2$ H_2SO_4 is added, the solution is boiled until CO_2 is expelled, (about 15 minutes) and the excess acid is titrated with $N/_2$ alkali.

1 c c. $N/_2$ = .0265 Na_2CO_3 = .0346 K_2CO_3.

The above methods for determining free and carbonate alkali are to be preferred to the methods as given by Bureau of Standards on account of the fact that if the soap contains both free alkali and unsaponified fat, these may combine with each other in the Bureau of Standards method. Under ordinary conditions the Bureau of Standards method is perfectly satisfactory. The directions specify 95% alcohol washing the carbonated alkali residue in the free and carbonated alkali determination and for making the stearic acid solution. Absolute alcohol is better, but 95% alcohol is sufficient for ordinary purposes. In the case of liquid soaps the soaps high in water, the sample must be dried before determining the free and carbonated alkali.

For routine analysis, the determinations of free caustic, carbonated, and combined alkali, by the methods given above, and the determination of salt by the Bureau of Standards methods, are sufficient. If it is desired to determine the total fatty acids, they can be easily determined as follows:

DETERMINATION OF TOTAL FATTY ACIDS

Five to 10 grams of soap are dissolved in hot water, decomposed with an excess H_2SO_4, and weighed amount of paraffin is added. The paraffin extracts the liberated fatty acids and the mixture gathers in a cake on top on cooling. This cake is dried between filter paper, weighed, and the fatty acids obtained by subtracting the weight of paraffin, provided the soap contains no unsaponified fat or waxes.

The determination of moisture is rather unsatisfactory, but results sufficiently accurate for ordinary purposes may be obtained as follows:

Have on hand a supply of oleic acid which has been dehydrated by heating to 120° C. and preserved in a well stoppered bottle. Weigh from 2 to 4 grams of soap in a porcelain evaporated dish, add 3 times its weight of the oleic acid, and weigh again. Heat the dish cautiously with a small flame until all the water is driven off, being careful not to heat enough to decompose the acid. At the moment when all the water is driven off, the soap forms a clear solution, provided there are no fillers present. Cool and weigh.

TOTAL AND COMBINED ALKALI

Weigh 5 grams of the sample into a large platinum dish. Ignite over the strong flame of the Bunsen burner, hastening the ignition by directing the flame of another burner into the dish. When the organic material ignites, allow it to burn freely to a charred black mass. Cool, sprinkle about 2 grams potassium chlorate evenly over the surface of the residue, cover with a watch glass, and holding the dish with a pair of tongs over a low flame, ignite gradually. Care must be taken that the action does not become violent and sputtering occur. When a white or yellowish white residue remains, ignite strongly to quiet fusion. Cool, rinse off the cover glass into the dish and wash down the sides of the dish with hot distilled water, using about 50 c. c. Add an excess of $N/_2$ sulphuric acid solution, 30 c. c. for common soap and 35 c. c. for toilet soap is usually sufficient. Boil for 5 minutes, when, if solution is complete and carbon dioxide expelled, titrate while still hot, the excess of acid with $N/_2$ potassium hydroxide solution, using phenolphthalein as an indicator.

The number of cubic centimeters of acid used, minus the number of c. c. of alkali used to titrate the excess, is the number of c. c. of acid used to neutralize the total alkali (free caustic, carbonated and combined) of the soap. The determination of free caustic and carbonated alkali is given in the Method of Determining Free Caustic and Carbonated Alkali in Soaps. The number of c. c. of acid used to neutralize the free caustic and carbonated alkali, subtracted from the number of c. c. of acid used to neutralize the total alkali in the sample, multiplied by the factor .31 gives the percentage of combined alkali, considered as Na_2O in the soap.

The factor is obtained by dividing the weight of the sodium oxide (Na_2O) equivalent of 1 c. c. of $N/_2$ sulphuric acid solution by the weight of the sample and multiplying by 100 ($.0155 \div 5 \times 100 = .31$)

In the case of liquid soaps the alkali is reported as K_2O instead of Na_2O. Instead of multiplying by .31 the factor is .47.

Throttle Packing

CONTAINS GREASE, ASH (Asbestos) AND GRAPHITE, WITH A RUBBER CORE

Determination of Grease and Ash: Remove rubber core, weigh, extract grease with gasoline, filter gasoline into a weighed beaker, evaporate off the gasoline and weigh the grease. Burn the residue in an electric muffle and weigh the ash. When the results are desired on the original sample, weigh up the sample before removing the rubber core.

There are other samples of this kind without the rubber core and consist of a plaited rope. On these, determine the ash and grease as above, and also the per cent. of water it will absorb. Namely: Weigh the sample unplaited, but do not unravel the threads. Place in cold distilled water for 48 hours. Remove and dry a few minutes to remove all the water from the surface, and weigh. The gain in weight, equals the water absorbed. Calculate the per cent. absorbed from the weight of the sample at the beginning.

Analysis of Lead Chromates, Chrome Greens and Prussian Blues

SCARLET LEAD CHROMATE

OIL

Make a gasoline extraction on 5 grams of the paste in a large mouth 150 c. c. flask. Weigh the flask; then weigh into it 5 grams of the paste. Into a tall weighed beaker, pour the gasoline from the flask after six hours. Repeat four times. Drive off the gasoline by placing the beaker on the hot plate. Weigh and calculate the per cent. of gasoline extract.

LEAD CHROMATE

One gram of pigment is weighed into a 250 c. c. beaker, 35 c. c. water + 15 c. c. acetic acid + 3 c. c. ammonium is added. Stand the beaker on the steam plate until all lead oxide is in solution, one-half hour being required. Remove the beaker and filter its contents through a weighed Gooch crucible. Heat the crucible until the lead chromate assumes a brick red color. Cool in the desiccator and weigh.

LEAD OXIDE

Weigh one gram pigment into a 250 c. c. beaker + 35 c. c. water + 15 c. c. hydrochloric acid is added. Place the beaker on the steam plate for one-half hour. Remove and titrate with potassium dichromate, using drop plate and silver nitrate as an indicator. When all lead is precipitated by the chromate, the excess of the chromate gives a brick red silver chromate on the drop plate.

Report:	Gasoline Extraction	?		
	Pigment	?	Lead Chromate	57-61%
	Volatile	?	Lead Oxide	38-47%
		100	(Desired)	

NOTE: The above method is to be used in checking scarlet lead chromate, which is bought under specification. Consequently, this method, which is somewhat empirical, is satisfactory for inspection purposes. The shade is by far the most important property, and samples may be rejected on shade alone, regardless of the analysis.

In cases where the analysis of an unknown lead chromate is desired, more elaborate methods must be used.

Chrome Greens

INSOLUBLE IMPURITIES

Ash .5 to 3 grams sample (depending on the amount of inert present). Cool and dissolve in 20 c. c. HCl by heating for about 15 minutes. Dilute with 100 c. c. of water, boil, filter, wash with hot water, ignite and weigh the insoluble impurities. If desirable, fuse with Na_2CO_3 and analyze impurities in the usual manner.

TOTAL LEAD

Add to filtrate 5 c. c. H_2SO_4 and evaporate to white fumes. Dilute with 50 c. c. water and filter. Heat precipitate and paper with 25 c. c. ammonium acetate (10 c. c. NH_4OH plus 15 c. c. acetic acid), dilute to about 300 c. c. and filter and wash the filter paper mass by shaking it up in 100 c. c. water and again filtering. Precipitate the lead with $K_2Cr_2O_7$ in the usual manner. $PbCrO_4 \times .6906 = PbO$.

CHROMIUM

Ash .5 to 3 grams sample (depending on amount of inert present). Dissolve in 20 c. c. HCl and evaporate in about 1 or 2 c. c. of liquid. Take up in 75 c. c. alcohol. (Lead compounds remain behind with insoluble impurities, Cr and Fe go into solution.) Filter. Evaporate Fe and Cr solution nearly to dryness, add 5 times the weight of $NaNO_3$, fuse over the blast in a nickel crucible. Dissolve in water and filter. To the filtrate containing the Cr as Na_2CrO_4 add 50 c. c. tartar emetic solution and then 30 c. c. of a saturated $NaHCO_3$ solution and titrate back with $N/_{10}$ iodine. Run a blank and estimate Cr by difference.

CALCIUM

Ash .5 gram to 3 grams (depending on the amount of Ca present). Cool and dissolve Ca by adding 10 c. c. acetic acid and 200 c. c. water. Boil and add a slight excess of $K_2Cr_2O_7$. Filter. Determine Ca by precipitation in the filtrate by NH_4OH and ammonium oxalate in the usual manner, disregarding the bichromate in solution, since it does not interfere with the precipitation of calcium oxalate. (Under ordinary conditions the Ca determination is unnecessary.)

IRON

Ash .5 to 5 grams (depending on amount of iron present), and dissolve in 20 c. c. HCl. Add $SnCl_2$ drop by drop until Fe is reduced to ferrous iron. (In the presence of large amounts of chromates this requires a little experience on account of the dark color of the chromium chloride.) Cool, dilute to 200 c. c., add 20 c. c. saturated

$HgCl_2$ all at once, let stand 2 minutes, titrate with $K_2Cr_2O_7$, using $K_3FeC_6N_6$ as outside indicator. In the absence of other iron compounds per cent. Fe times 3 equals approximately per cent Prussian Blue.

SULPHATES

Ignite sample in a casserole, cool, dissolve in HCl, filter, and precipitate sulphates with barium chloride in the usual manner.

NOTES

"C. P." Chrome Yellows are those that contain only lead chromate, lead sulphate, lead oxide, lead carbonate, or other lead compounds. "C. P." Chrome Greens are mixtures of "C. P." Chrome Yellows and Prussian Blue or Chinese Blue. Since this mixture explodes when heated, it is necessary to brind a sample of "C. P." Chrome Green with from 2 to 5 times its weight of barium sulphate before igniting it to prevent it from exploding. If the Chrome Green already contains considerable inert pigment, the addition of barium sulphate is unnecessary.

Prussian Blue must be ignited before solution for analysis of iron, but it is not explosive under ordinary conditions.

The above methods of analysis will give satisfactory results on Chrome Yellows, Scarlet Chromes, Chrome Greens, Prussian Blues, and Chinese Blues, regardless of whether they are present separately or combined with one another.

Methods for the Examination of Soda Ash

The density, moisture, direct carbon dioxide, and sodium oxide are determined by chemical analysis. From the results thus obtained, the carbon dioxide, sodium carbonate, dry sodium carbonate and 58 per cent. sodium carbonate are calculated.

DENSITY

Weigh 30 grams of the sample. Pour into a 100 c. c. graduated cylinder. Pour it in loosely and do not pack. The number of c. c. this weight of soda ash occupies determines the acceptance or rejection of the shipment.

MOISTURE

Weigh 5 grams of sample on a watch glass and place it on the hot plate (steam heated plate) for about 5 hours, or until the weight becomes constant. Cool in a desiccator and weigh. The loss in weight is considered as moisture.

CARBON DIOXIDE

One gram of sample is weighed into the evolution flask of the carbon dioxide apparatus, 50 c. c. of water is added and the carbon dioxide is evolved by adding slowly from a dropping funnel, 25 c. c. of dilute sulphuric acid (1 to 4.) The evolved carbon dioxide is passed through concentrated sulphuric acid and calcium chloride to dry it and is then absorbed in a tared Geissler potash bulb with a calcium chloride prolong. The potash solution used in this bulb is made of such concentration that it has a Sp. Gr. of 1.27 (about a pound of C. P. potash to 1 quart of water will give approximately the concentration desired.) The increase in weight of the absorption apparatus is, of course, due to the absorption of carbon dioxide, and since one gram sample was taken, multiply this weight in grams by 100 will give the percentage of direct carbon dioxide.

SODIUM OXIDE

Weigh 1 gram of the sample into a 250 c. c. Erlemeyer flask. Add 150 c. c. water and dissolve, boiling it about 5 minutes. When all is in solution, filter off any mineral matter present and weigh. Cool the flask and contents in an ice bath and when cold, titrate with half normal sulphuric acid using Methyl Orange as an indicator. Run a duplicate and a blank the same way. This number of c. c. of half normal

sulphuric acid required to react with 1 gram of soda ash is the basis from which are calculated the following:

(1) Sodium Carbonate.
(2) Sodium Oxide.
(3) Carbon Dioxide (Indirect).
(4) 58% Sodium Carbonate.
(5) Dry Sodium Carbonate.

Any mineral matter present, may be detected.

SODIUM CARBONATE

To calculate the sodium carbonate the value of 1 c. c. of the $N/2$ sulphuric acid in terms of C. P. sodium carbonate must be known. This is determined as follows: Heat to dull redness in a clean tared platinum crucible about 1.2 grams of C. P. sodium carbonate. Cool crucible and contents in a desiccator. Weigh accurately and note the exact weight of dried C. P. sodium carbonate. Dissolve the mass by placing crucible and contents in hot distilled water. Boil until solution is complete; then cool the solution in an ice bath and when cold titrate with half normal sulphuric acid, using methyl orange as an indicator. The number of c. c. of acid required to react with sodium carbonate divided into the weight of the sodium carbonate used gives the value of 1 c. c. of the acid in grams of C. P. sodium carbonate. This result should be checked several times. Having now combined this factor, to determine the amount of sodium carbonate in the soda ash, multiply the number of c. c. of half normal sulphuric acid required to react with 1 gram of soda ash by factor, and, since 1 gram of sample was used, the result thus obtained, multiplied by 100 will give the per cent. of sodium carbonate in the soda ash.

SODIUM OXIDE

Multiply the grams of sodium carbonate determined as above by the factor 0.5849 (as required by the specifications) and the result thus obtained by 100 for the per cent. of sodium oxide.

CARBON DIOXIDE (Indirect)

Multiply weight of sodium carbonate in grams found as above described, by the factor 0.4151 and this result by 100 for the per cent. of carbon dioxide.

58% SODIUM CARBONATE

Divide the weight of sodium oxide in grams, as calculated above by .58 and multiply this result by 100.

SODIUM CARBONATE IN DRY SODA ASH

Divide the actual sodium carbonate by the moisture free soda ash.

Calibrating Thermometers

The simplest method of calibrating thermometers is by comparing them with other thermometers which have previously been standardized. The thermometer to be calibrated should be placed very close to the standard thermometer in a bath of water, oil, or other suitable liquid whose temperature can be varied. The liquid should be thoroughly stirred and the comparative readings of the two thermometers taken at different temperatures. For very accurate work it is necessary to make a large number of readings and average the results. Most thermometers are graduated for total immersion; that is, the graduations are correct only when the bulb and stem of the thermometer are both immersed in a liquid or other material at the temperature that is being measured. If the bulb is at one temperature and the stem is at another temperature, it will be necessary to apply the "stem correction." Under extreme conditions this "stem correction" may amount to 10 or 15 degrees. The "stem correction" equals .00016° C. (.000089° F.) $\times N(T\text{-}t)$. $N =$ number of degrees emergent from bath. $T =$ temperature of bath. $t =$ average temperature of the emergent stem.

Example: The observed temperature is 50° C. and the thermometer is immersed up to the 0° C. of the mercury column projected oupt into the air and the average temperature of the emergent stem is found to be 20° C.

$$\text{The stem correction} = .00016 \times 50(50\text{-}20)$$
$$= .240$$

Titre Test

The determination of the titre of a fat is to ascertain the temperature, expressed in degrees Centigrade, at which the free fatty acids extracted form a fat or solidify. This test is also called the hardness, or freezing point test. Animal fats consist largely of glycerides of oleic, stearic and palmatic acids. Almost every animal fat contains a small percentage of free fatty acids, together with a smaller or larger percentage of impurities.

The determination of a solidifying temperature of the fatty acids of a given fat requires first of all that the fatty acid be extracted from the fat and separated from the glycerine, combined with which they form as glycerols the bulk of the fat. In order to obtain the free fatty acids it is necessary to follow a round about way, as it is impossible to separate the fatty acids from the glycerine directly, except by subjecting the fat to a degree of heat and pressure and the action of a relatively smaller chemical which is apt to chance a change in the free fatty acid.

When an animal fat is treated and heated with a sufficient amount of some strong alkali, such as sodium hydrate, the glycerides are decomposed, the alkali combining with the free acids, forming soap.

Oleic Glyceride Sodium Oleicate Glycerine

$$C_3H_5(C_{18}H_{33}O_2)_3+3NoH=3NaC_{18}H_{33}O_2+C_3H_5(OH)_3$$

Palmatic Glyceride Sodium Palmatate

$$C_3H_5(C_{16}H_{31}O_2)_3+3NoH=3NaC_{16}H_{31}O_2+C_3H_5(OH)_3$$

Stearic Glyceride Sodium Stearate

$$C_3H_5(C_{18}H_{35}O_2)_3+3NoH=3NaC_{18}H_{35}O_2+C_3H_5(OH)_3$$

The soap formed, when treated with a stronger mineral acid, as HCl or H_2SO_4, is decomposed, giving the free fatty acid as an oily layer floating on the top of the hot aqueous solution of the mineral acid and its salts. Remove the lower layer, wash free of mineral acids and its salts with water in a separating funnel, keep hot and do not let the fat cool or solidify, dry to remove water, and the free fatty acids are in a pure state. Use NaOH with alcohol to insure complete saponification (40 c. c. NaOH + 33 c. c. alcohol) of the glycerides. The alcohol must be completely removed before treating with the mineral acid.

Sp. Gr. 1.33=30% NaOH 300 grams to 1 liter water.
Sp. Gr. 1.14=20% H_2SO_4.

DALICAN SAPONIFICATION METHOD

Fifty grams of tallow is heated to 396 degrees F. in an enameled iron dish or porcelain casserole. Add 40 c. c. NaOH+33 c. c. alcohol, with constant stirring. The heat of the dish is enough to drive off most of the alcohol and give an almost dry, crumbly soap, which is then dissolved in boiling water and decomposed with 19 c. c. H_2SO_4 (Sp. Gr. 1.84) dissolved in 100 c. c. water. Heat on the steam plate until the fatty acids appear on the top as a clear oily liquid. Transfer it to a separatory funnel, take off the H_2SO_4 layer, wash twice with 2 per cent. hot H_2SO_4, then with hot water, until the water gives no acid reaction with litmus. Transfer fatty acids to a 250 c. c. beaker and dry at 100 degrees C. for 2 hours.

In the determination proper, a thin wall test tube is used, (1¼" x 6".) This is fixed by means of a cork into a bottle. A centigrade thermometer graduated in tenths of a degree is fixed by means of a cork into a test tube, which should be loose enough to permit easy stirring. The test tube is filled nearly full of the dry fatty acids. The liquid is then stirred until quite opaque, and partly solidified. After stirring from 10 to 12 times, the thermometer is allowed to stand at rest. The latent heat liberated from the solidifying acids causes a rise in temperature, and the highest temperature is taken as the solidifying point or titre of the fatty acid. The mercury stands at this point for several minutes. Each titre test should be repeated and the difference should not be greater than .1 of a degree.

42° Centigrade is the low limit for tallow.

Oil Analysis

The routine work on oils consists of the analysis and tests on all kinds of oils bought on specification. These oils are for lubrication, painting and illumination. When oils are bought through contract, a sample of every shipment is tested to see that it is in accordance with the sample submitted before the signing of the contract, as representing the material that would be furnished. Fuel oil of various burning points is used. The burning point of samples of these oils is determined and this test is used in deciding how the oil will be used.

The lubricating oils are known as coach, car and engine, both summer and winter grades, 400 degree coach, well, paraffine, 500 degree, valve, superheater valve, turbine, air compressor and Extra No. 1 Lard oil.

The oils for painting, or the drying oils, are both raw and boiled linseed oil.

The oils for illumination are 150 degree, 300 degree and signal oil. The signal oil is made of one part prime lard oil and two parts 300 degree oil.

A chart is herewith attached giving the routine of these oils (page 60).

The specific gravity of the different oils is taken as described in "The Method of Determining the Specific Gravity of Oils and Other Liquids," using the most applicable method of those given, except that in the case of superheater valve oil this test is made at 100 degrees Fahrenheit.

The cold test and chilling points are obtained as described in "The Method of Taking Cold Test and Chilling Points of Oils and Other Liquids."

The flashing and burning points are obtained as described in "The Method of Determining the Flashing and Burning Points of Combustible Liquids."

The KOH test is made on 150 degree and 300 degree oils to see if they have been contaminated with fat oil. Test: To a 4-oz. bottle 2/3 full of distilled water, add about 25 c. c. of the sample to be tested, and a few drops of potassium hydroxide solution (13 degrees Beaume.)

Shake, and on standing 10 minutes note if a white soapy ring, indicating fat oil, is formed between the oil and the water solution.

To determine the approximate percentage of fat oil, the sample must be saponified as described later.

The tar test is made on all dark colored petroleum lubricating oils. Test: To 95 c. c. of gasoline in a 100 c. c. graduated glass stoppered cylinder, add 5 c. c. of the sample to be tested. Shake, and on standing 10 minutes, note the presence of flocculent or tarry matter.

The free acids are determined in valve, superheater valve, lard, turbine and signal. This method is described in "The Method of Determining Free Acids in Oil and Tallow."

Saponification of Compounded Lubricating Oils: Weigh into a 250 c. c. Erlemeyer flask 5 grams of the sample, add 50 c. c. of alcoholic potash and 50 c. c. of benzole. Stopper the flask with a reflux condenser and place on the steam plate for four hours. Titrate with half normal hydrochloric acid solution, using phenolphthalein as an indicator. A blank determination is run under the same conditions. The difference between the number of cubic centimeters of half normal hydrochloric acid solution used for the blank determination, and the sample is equivalent to the number of cubic centimeters of a half normal potassium hydroxide solution needed to saponify the fat oil. This number of cubic centimeters multiplied by .02805 (grams potassium hydroxide in 1 c. c.) and divided by the weight of the sample, and .193 (saponification number of the oils) and multiplied by 100 gives the percentage of fat oil in the sample. The alcoholic potash solution is made by dissolving 32.17 grams of potassium hydroxide in 1 liter of ethyl alcohol. Only the clear solution is used.

If a large percentage of mineral oil is present in the sample, 10 grams should be used for this determination.

When lard and linseed oils are saponified, benzol is not used, and the time taken must be sufficient for complete saponification.

Maumene Test: Stir together with a thermometer in a beaker 20 c. c. of a lard or signal oil sample, or 5 c. c. raw or boiled linseed oil, fish or bean oil and 10 c. c. of oil of vitriol and note the rise in temperature at its highest point. The oil of vitriol must be standardized as follows: 30 c. c. of distilled water and 10 c. c. of oil of vitriol must be mixed in a beaker and give a rise in temperature of 100 degrees Fahrenheit. In starting the test and standardization, the samples and reagents must be at a temperature of 80 degrees Fahrenheit. The beaker must be of glass and weigh 19 to 20 grams. Satisfactory oil should give a rise in temperature closely approximating the following:

5 c. c. raw linseed,	10 c. c. acid, from 80 Deg. F. to 186 Deg. F.
5 c. c. boiled linseed,	10 c. c. acid, from 80 Deg. F. to 186 Deg. F.
5 c. c. fish,	10 c. c. acid, from 80 Deg. F. to 185 Deg. F.
5 c. c. Soya bean,	10 c. c. acid, from 80 Deg. F. to 170 Deg. F.
20 c. c. signal (contract),	10 c. c. acid, from 80 Deg. F. to 113 Deg. F.
20 c. c. Extra No. 1 lard	10 c. c. acid, from 80 Deg. F. to 155 Deg. F.
20 c. c. prime lard	10 c. c. acid, from 80 Deg. F. to 157 Deg. F.

Carbonization test on Air Compressor Oils: Weigh 13 grams of the sample in a tared platinum dish, place in a double lined copper oven which has been heated to 425 degrees Fahrenheit and maintain at this temperature for 6 hours consecutively. The oven should be connected with the suction so that the vapors can be carried off without igniting. Cool and weigh. The loss in weight divided by the weight of the sample and multiplied by 100 gives the percentage of volatile material. Dissolve the oil in gasoline and filter through a Gooch crucible which has been prepared with a tight asbestos felt. Dry and weigh. Ignite the residue in the crucible, cool and weigh. The difference between these two weights, divided by the weight of the sample and multiplied by 100, gives the relative amount of carbonization.

The 24 hours burning test is made on the signal oil only. A hand lantern such as trainmen use is filled with oil and burned for 24 hours, without moving the wick. Note if the oil burns with a free flame continuously, and if any crust is formed.

Silver nitrate test is made on prime lard oil.

The nitrate of silver test is as follows: Have ready a solution of nitrate of silver in alcohol and ether, made on the following formula:

Nitrate of Silver,	1 gram
Alcohol,	200 grams
Ether,	40 grams

After the ingredients are mixed and dissolved, allow the solution to stand in the sun or in diffused light until it has become perfectly clear; it is then ready for use and should be kept in a dimly lighted place and tightly corked.

Into a 50 cubic centimeter test tube, put 10 cubic centimeters of the oil to be tested (which should have been previously filtered through washed filter paper), and 5 cubic centimeters of the above solution, shake thoroughly and heat in a vessel of boiling water fifteen minutes with occasional shaking. Satisfactory oil shows no change of color under this test.

The Halphen Test is a more delicate test for cotton seed oil. The reagent is a mixture of equal volumes of amyl alcohol and carbon bisulphide, in which 1% of sulphur has been dissolved. About 5 c. c. of the melted fat is mixed with an equal volume of this reagent in a test tube. The tube is loosely stoppered with cotton and heated in a boiling saturated salt solution 15 minutes. In the absence of cotton seed oil little or no color is developed, but in the presence of cotton seed oil a deep red color is produced.

The 450 degree test is made on 300 degree oil only. Test: Heat about 50 c. c. of the sample in a 100 c. c. beaker to 450 degrees Fahrenheit on a sand bath. Note if precipitation of carbon has occured and if not the oil is considered to have passed the test satisfactorily.

VISCOSITY

Viscosity is the degree of fluidity of an oil, or its internal friction. It is independent of the specific gravity of the oil, although this, in the pipette instruments, influences the time of efflux. Within certain limits, it may be taken as a measure of the value of oil as a lubricant, by comparing the viscosity of the oil under examination, with that of other oils which have been found to yield good results in practice.

The instruments employed for its determination may be divided into two classes; pipette viscosimeter, giving the time of efflux, as those of Engler, Saybolt, and others, and torsion viscosimeters, giving the retardation due to oil, those of Napier and Doolittle.

In expressing viscosity, consequently, it is necessary to give the name of the instrument with which it is determined. It is sometimes expressed as specific viscosity; that is, the time of efflux of the oil, divided by the time of efflux of water, but specific viscosity, Engler, is not the same figure as specific viscosity, Saybolt. Besides this manner of expressing viscosity, it is occasionally measured in absolute (C. G. S.) units, or dynes. This is possible when the diameter of the orifice, its length, the quantity and specific gravity of the oil, its time of efflux and change of head are known. Where it is impracticable to determine all this data by direct measurements, the reading of the viscosimeter may be changed into dynes by determining the viscosity in seconds, of standard solutions of glycerine, the viscosity of these being determined in dynes from tables of physical constants.

The Engler Viscosimeter used in this laboratory may be described as follows: The apparatus consists of a flat, brass cylindrical vessel, 106 m. m. in diameter and about 62 m. m. deep, provided with a jet 2.9 m. m. in diameter at the top and 2.8 m. m. at the bottom, and exactly 20 m. m. long. This vessel is gold-plated inside and the jet is of platinum. It is surrounded with a bath either of water or oil, provided with a stirrer and heated by a ring burner. The jet is closed by a wooden valve passing through the cover, and a thermometer to show the temperature of the oil. Three studs show the height to which the inner vessel is filled and at the same time, when it is level. The oil is ordinarily discharged into the 200 c. c. flask, although in case the oil or time be limited, 100 or 50 c. c. may be used and the time of efflux multiplied by a suitable factor. The instrument is standardized with water, 200 c. c. of which at 70 degrees Fahrenheit should run out in 52 seconds. The instrument is thoroughly cleaned with alcohol and ether if necessary, and dried; any suspended matter is removed from the oil which is poured into it up to the level of the studs, stirred until 70 degrees Fahrenheit is reached and the bath adjusted to the same temperature.

The flask is placed beneath the orifice, the plug raised and the time required for 200 c. c. of oil to flow out is noted; this is divided by the water value of the viscosimeter and gives the relative, or specific viscosity. Example: The time taken for 200 c. c. of the sample to flow out is 4 hours and 50 minutes, 290 minutes, divided by 52, equals 5.57, the specified viscosity.

Conradson Carbon Test

METHOD

Put 10 grams of the oil to be tested into the porcelain crucible and place the latter in the Skidmore crucible. Place the Skidmore crucible in the exact center of the iron crucible and put on the covers of the Skidmore and iron crucibles. Set the apparatus us as indicated in Fig. 1.

Apply heat with a Bunsen or other burner using a high flame around the large crucible. Continue heating intensely until oil vapors begin to ignite over the crucible. Then decrease the heat so that the oil vapor (flame) comes off at a uniform rate. (The oil vapor flame should not extend over two inches above the hood.) After the vapor ceases to come off, increase the heat as at start and maintain for five minutes, making the lower part of the iron crucible red hot. Allow the apparatus to cool somewhat

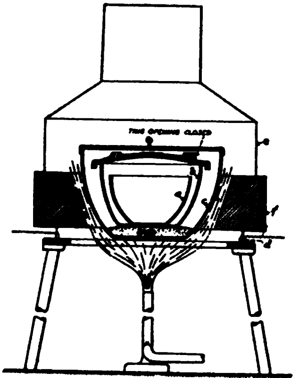


FIG 1-APPARATUS FOR CONRADSON CARBON TEST.

Fig. 1.

before uncovering the crucible. Remove the porcelain crucible, cool in a desiccator and weigh.

The entire process should be completed in about one-half hour when the heat is properly regulated. The time varies somewhat with the kind of oil tested; i. e., a very thin oil with a low flash-point will not take as long a time as a heavy thick oil with a high flash-point.

APPARATUS

Fig. 1 shows the arrangement of the apparatus for this test. It consists of the following:

(*a*) Porcelain crucible; wide form, completely glazed, 25 to 26 c. c. capacity, 46 m. m. in diameter.

(*b*) Skidmore iron crucible; 45 c. c. (1½ oz.) capacity, 65 m. m. in diameter, 37 to 39 m. m. high with cover, without delivery tubes and one opening closed.

(*c*) Wrought iron crucible with cover, about 180 c. c. capacity, 80 m. m. in diameter, 58 to 60 m. m. high. About 10 m. m. of sand placed in bottom to bring Skidmore crucible nearly flush with the top.

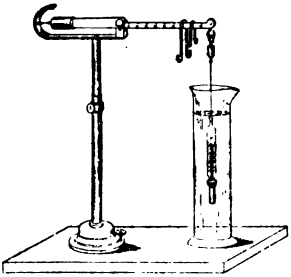
(*d*) Triangle; pipe stem covered, projection on side to allow flame to reach all sides of the crucible.

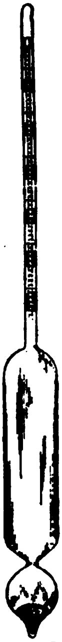
(*e*) Sheet iron or asbestos hood provided with a chimney about 2 to 2½ inches high and 2⅛ to 2¼ inches in diameter. This serves to distribute the heat uniformly.

(*f*) Asbestos block.

Method of Determining Specific Gravity of Oils and Other Liquids

OPERATION





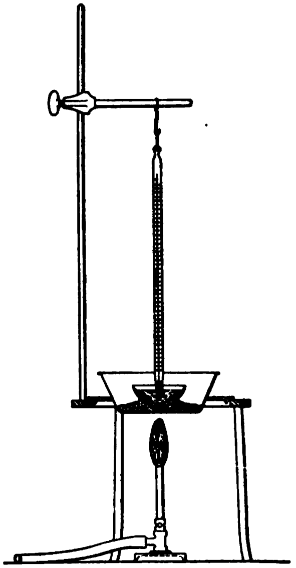
1. When using the hydrometer—applicable to all liquids, whether transparent or not, except such as are too viscous —fill the hydrometer jar to a convenient height with the liquid to be tested, introduce the hydrometer, and along side of it a delicate thermometer sufficiently long to reach the bottom of the jar, and be used as a stirring rod. Stir with the thermometer, disregarding the hydrometer for the moment, and taking pains to have the thermometer reach to the bottom of the liquid, until the reading of the thermometer becomes constant. Read the thermometer and withdraw it from the liquid. Raise the hydrometer and wipe the stem clean, nearly down to the bulb, then carefully allow it to sink in the liquid. This operation must be managed so that the stem of the hydrometer will not be wetted above the point corresponding to the gravity of the liquid. Read now the mark on the stem corresponding to the level of the liquid. The scale the hydrometer is graduated in, corresponding to that temperature, and this reading when corrected for temperature, as provided under "calculations" becomes the reading to be used. The method described above is that applicable to the petroleum products. When other liquids are tested with the hydrometer, the temperature must be made 60° F. before reading the hydrometer, and no corrections are necessary.

II. When using the Westpal balance—applicable to all liquids, except such as are viscous, but works best with transparent liquids—set up the balance on a level place, by placing the beam in position and hanging the plummet on the hook provided for it. Now adjust with the adjusting screw in the foot until the pointers are exactly opposite each other, cool the liquid to be tested to a point a little lower than that at which the balance was graduated, fill the jar with enough of the liquid, to a little more than cover the plummet, and place the jar in position. Allow the temperature of the liquid to raise with occasional stirring, until the reading of the thermometer in the plummet is that at which the balance was graduated. Now hang the weights on the beam at the notches provided for them, until the pointers are again exactly opposite each other. If two belong on the same notch, hang the second on the hook of the first. Read the marks on the beam where the weights hang, and set down the figures by the side of each other, putting the figure under the heaviest weight at the left hand, that under the next heaviest, next, and so on. The result will be the specific gravity expressed in decimals when the liquid is lighter than water. For liquids heavier than water it will be found that one of the heaviest weights will hang on the hook along with the plummet, the others being distributed along the beam. The reading of the heaviest weight will then be 1, which must be followed by a decimal point, the other readings following the order of the size of the weights, as above described, being placed after this decimal point. In case the specific gravity of the liquid in question is 2 or above, or 3 or above, two or three of the heaviest weights, as the case may be, will hang on the hook, the others being distributed along the beam. When the liquid to be tested is opaque, so that the reading of the thermometer in the plummet cannot be made, it is essential to have an additional thermometer to use in determining the temperature. .This additional thermometer should, of course, be compared with the one in the plummet.

III. When weighing a known volume of the liquid and comparing it with the weight of the same volume of water—applicable to all liquids—weigh any convenient graduated vessel with a thermometer in it, cool the liquid to be tested to a temperature a little below 60° F., pour into the weighed graduate any convenient measurable amount and weigh. Clean the graduate and repeat the operation with distilled water, using the same volume as before. The ratio of the two weights, using the weight of the water as divisor, is the specific gravity of the liquid.

Methods of Determining Flashing and Burning Points of Combustible Liquids

OPERATION



Have ready the apparatus shown in the cut. Light the lamp, put it under the centre of the porcelain dish, adjust it as previous experience has indicated is necessary to secure the proper rate of heating, and allow the apparatus to get warm. Fill the porcelain dish to within about one-fourth of an inch of the top with the liquid to be tested, adjust the thermometer in the centre of the liquid, and so as to reach nearly to the bottom of it, but not quite touching the dish. Adjust the flame so that the temperature of the liquid in the dish rises at the rate required for the liquid being tested, and when the temperature reaches a desired point, apply the test flame by passing it slowly, entirely across the dish, about a half an inch above the level of the liquid and just in front of the thermometer. Allow the liquid to rise in temperature until another testing point is reached and then apply the test flame again in the same manner. Proceed in this way until the vapor from the liquid above it ignites with a slight flash. The temperature shown by the thermometer when this is the case is the flashing point of the liquid. Continue the heating and testing in the same manner until a point is reached where the liquid takes fire. The reading of the thermometer when this is the case is the burning point of the liquid. Remove the thermometer, blow out the flame, or smother it by sliding a watch glass over the top of the porcelain dish, empty the dish and wipe it out, when the apparatus is ready for another test.

Method of Taking Cold Test and Chilling Points of Oils and Other Liquids

OPERATION

For cold test, put about 1 oz. of the liquid to be tested into a common 4 oz. sample bottle, and place a short stout thermometer in it. Then place the bottle in a situation where the liquid will become frozen, using for this purpose a freezing mixture if necessary. When the liquid has become solid throughout, remove from the cold and allow the liquid to soften, thoroughly stirring and mixing at the same time, by means of the thermometer, until the mass will run from one end of the bottle to the other. Now grasp the bottle by the neck, having in the same hand a little waste or a towel, which encloses the thermometer, withdraw the thermometer through the waste, to wipe it far enough to see the mercury, and the temperature. The reading is the cold test of the liquid.

For chilling point, where it is desired to know *whether the liquid remains clear at any given temperature.* Use the same bottle, amount of liquid, and thermometer as for the cold test. Expose the liquid to a given temperature, or a little below, stir it with the thermometer occasionally, until the temperature of the whole liquid becomes that desired,. then allow to stand for the time specified, at the end of which observe whether the liquid remains transparent and free from flocks or scales of congealed constituents. If so, it stands the test.

For chilling point, where it is desired to know *at what temperature flocks or scales of congealed constituents or more or less opacity takes place.* Use bottle, amount of liquid, and thermometer as before. Expose to a temperature 5° colder than that at which the liquid is perfectly clear and free from flocks or scales, and allow the temperature to fall to that of the source of cold, with constant watching and occasional stirring. Lower the source of cold 5° more and treat as before. Proceed in this way until opacity or flocks or scales begin to show. The reading of the thermometer when this is the case is the temperature sought.

Method of Determining Free Acids in Oils and Tallow

MATERIALS REQUIRED

½ dozen 4 ounce Sample Bottles.

3 10 Cubic Centimeter Pipettes, or if desired a balance weighing milligrams.

1 30 C. C. Burette, graduated to tenths, (Burette Holder if desired) with Pinch Cock and Delivery Tube.

2 oz. Alcoholic Solution of Turmeric.

2 qt. 95 per cent. Alcohol to which ⅛ oz. dry Carbonate of Soda has been added and thoroughly shaken.

1 qt. Caustic Potash Solution, of such strength that 31½ Cubic Centimeters exactly neutralize 5 Cubic Centimeters of a mixture of Sulphuric Acid and Water, which contains 49 milligrams $H_2\ SO_4$ per Cubic Centimeter.

OPERATION

Take about 2 ounces of the clear Alcohol and add a few drops of the Turmeric Solution, which should color the Alcohol red, warm to about 150° Fahrenheit, then add 8.9 grams of the oil to be tested and shake thoroughly. The color of the solution changes to yellow. Fill the Burette to the top of the graduation with Caustic Potash Solution, and then run this solution from the Burette into the bottle little at a time, with frequent shaking, until the color changes to red again. The red color must remain after the last thorough shaking. Now read off how many cubic centimeters and tenths of the Caustic Potash Solution have been used, and this figure gives directly the acid number of the oil.

To determine the Free Acid in Tallow, everything is done exactly as above described, except that the tallow is melted before it is added to the alcohol.

Ten Cubic Centimeters of Extra Lard Oil, at ordinary temperatures, and the same amount of melted Tallow at 100° Fahrenheit, weigh almost 8.9 grams. In ordinary work therefore it will probably not be necessary to weigh the oil or tallow. Measurement with a 10 Cubic Centimeter Pipette, will usually be sufficiently accurate, provided the Pipette is warmed to about 250° Fahrenheit, and allowed to drain, the last drops being blown out. In case of dispute, however, the balance must be used.

Method of Determining Iodine Value of Linseed Oil

EXPLANATORY

The iodine value is the percentage of iodine absorbed by an oil, or the number of grams of iodine absorbed by 100 grams of oil. It is a measure of the degree of unsaturation of the fatty acids of the oil. The method depends on the fact that different oils absorb different amounts of iodine, which amounts are, with a few exceptions, nearly proportional to the drying power of the oils. The iodine value is considered most valuable as a single method for testing the purity of linseed oil. The iodine number varies somewhat, but usually runs about 180 for raw linseed oil. According to the report of the American Society for Testing Materials, the iodine value of a raw linseed oil from North American seed, should be at least 178.

APPARATUS AND REAGENTS

Flasks, beakers, etc., are fully described under "Operation."

IODINE SOLUTION (Hanus:) Weight 13.2 grams C. P. iodine into a 1000 c. c. flask and add 1000 c. c. glacial acetic acid. The iodine will go into solution easier if only about 50 c. c. of the acid be added to the iodine and allowed to stand over night; then add the remainder of the glacial acetic acid. After the iodine is entirely dissolved, add 3 c. c. bromine, and shake.

POTASSIUM IODIDE SOLUTION: Use 150 grams C. P. potassium iodide to make a liter of solution with distilled water.

SODIUM THIOSULPHATE SOLUTION: Use 24.8 grams of sodium thiosulphate to make a liter of solution with distilled water.

STARCH PASTE: Use 5 grams of starch per liter. Triturate the starch into a smooth paste with about ten c. c. cold water. Then bring a liter of water to boiling and slowly add the starch paste with stirring. Starch paste should not be used that is over a week old, as it starts to mold. (Treadwell.)

DECINORMAL POTASSIUM DICHROMATE SOLUTION: Dissolve 4.908 grams of C. P. potassium dichromate in distilled water and make the volume up to one liter, at the temperature at which the titrations are to be made. The dichromate solution should be checked against an iron wire of known composition.

OPERATION

Weigh 0.2 grams of sample of oil onto a small watch glass, and place it in a wide mouth bottle with a ground glass stopper. Add

10 c. c. of chloroform and 25 c. c. iodine solution. At the same time run a blank, using 10 c. c. of chloroform and 25 c. c. iodine solution. The iodine is best measured with a pipette, but care must be taken to add exactly the same amount to the sample as is used for the blank. Stopper the bottle and allow to stand in a dark place for 45 minutes, agitating the contents at frequent intervals.

In the meantime, standardize the sodium thiosulphate solution, as follows: Into a 250 c. c. flask, with a ground glass stopper, run 25 c. c. of an exactly decinormal solution of potassium dichromate, add 10 c. c. potassium iodide solution and 5 c. c. C. P. hydrochloric acid, 1.19 Sp. Gr. Titrate with sodium thiosulphate, until the yellow color of the iodine has almost disappeared, now add several drops of fresh starch paste and continue the titration until the blue color changes to a sea green, due to chromium trichloride. The end point should be accurate to 1 drop.

Atomic weight of iodine—126.92.

1 c. c. $N/_{10}$ iodine solution contains .012692 grams of iodine.

1 c. c. $N/_{10}$ potassium dichromate solution is equivalent to 1 c. c. iodine solution.

Hence 25 c. c. $N/_{10}$ potassium dichromate solution are equal to 2" times .012692 grams=.3173 grams iodine, a constant factor.

.3173 divided by the number c. c. sodium thiosulphate solution equals the number of grams of iodine equivalent to 1 c. c. of the sodium thiosulphate solution, or factor.

If the sodium thiosulphate solution were exactly tenth normal this standardization would be unnecessary, but sodium thiosulphate does not make a stable solution, and hence, it is necessary to restandardize it each time a determination is made. The value of 1 c. c. sodium thiosulphate solution should, however, approximate .012692 grams of iodine.

When the sample and the blank have stood 45 minutes, add to each, 10 c. c. of the potassium iodide solution and 100 c. c. of water. Titrate to faint yellow with the sodium thiosulphate solution which has already been standardized. Then add starch paste to the sample and titrate to water white. From the number of c. c. used to titrate the blank, subtract the number of c. c. used to titrate the sample and the result will be the number of c. c. of sodium thiosulphate solution which would be required to titrate the iodine absorbed by the oil. This number of c. c. of sodium thiosulphate solution times the factor (for iodine equivalent of 1 c. c. sodium thiosulphate solution) will give the weight of iodine absorbed by the oil. This weight divided by the weight of the sample, multiplied by 100 will give the idoline value.

SAMPLE CALCULATION

Number of c. c. of sodium thiosulphate solution to titrate 25 c. c. of potassium solution: 25.2 c. c.

.3173 divided by 25.2 equals .012591 (factor.)

Number of c. c. of sodium thiosulphate solution to titrate blank, 52.7 c. c.

Number of c. c. of sodium thiosulphate solution to titrate sample, 27.9 c. c.

52.7 minus 27.9 equals 24.8 c. c. of sodium thiosulphate solution, which would be required to titrate iodine absorbed by sample of oil.

.012591 times 24.8 equals .312256.

.312256 divided by .2 equals 1.5612 grams iodine absorbed by 1 gram of oil.

1.5612 times 100 equals 156.12 per cent. of iodine absorbed, or iodine number.

CHART OF WORK ON ALL SHIPMENT OILS

Kind of Oil	Gravity at 60° F. Beaume	Flash Deg. E.	Burn Deg. F.	Cold Test	Examined at Deg. F.	KOH Test	Tar Test	Saponification	Free Acid	Maumene	24 hr. Burn	Silver Nitrate	450° Test	Viscosity Deg.	Volatile F.	Pb & Mn	Spec'n Number
150°	47	(130	(151	Ice & Salt	0	taken	Not taken	Not taken	Not taken	Not taken	Not taken	Not taken	Not taken	Not taken	Not taken	Not taken	20-A
300°	39	(256	(298	Plain Ice	32	"	"	"	"	"	"	"	taken	"	"	"	20-A
Coach	W 26.5 S 25-26	W 221 S 312	Not taken	Ice & Salt	W 0-10 S 10-20	Not taken	taken	"	"	"	"	"	Not taken	"	"	"	Contract
Car	W 28.4 S 27.0	W 214 S 312	"	"	W 0-10 S 10-20	"	"	"	"	"	"	"	"	"	"	"	Contract
Engine	W 27.2 S 26.0	W 221 S 312	"	"	W 0-10 S 10-20	"	"	"	"	"	"	"	"	"	"	"	Contract
400° Coach	25-28	300-403	"	"	20	"	"	"	"	"	"	"	"	"	"	"	Contract
Well	28-0	382	"	"	10	"	"	"	"	"	"	"	"	"	"	"	20-A
Fuel	25-34	200	242	"	0-10	"	"	"	"	"	"	"	"	"	"	"	None
Signal	34-3	277	305	Not taken	Not taken	"	Not taken	"	taken	taken	taken	"	"	"	"	"	Contract
Paraffine	24-35	347	Not taken	Ice & Salt	0-5	"	"	"	Not taken	Not taken	Not taken	"	"	"	"	"	20-A
Wood Preserver	103-105	(172	(200	"	10-20	"	n)10%	"	"	"	"	"	"	"	"	"	35-A
Raw Linseed	Not Taken	)500	Not taken	Not taken	Not taken	"	Not taken	"	"	taken	"	"	"	"	taken	"	Monthly Sample
Boiled Linseed	"	)500	"	"	"	"	"	"	"	"	"	"	"	"	"	taken	Monthly Sample
500°	"	)500	"	"	"	"	taken	"	"	Not taken	"	"	"	"	Not taken	Not taken	20-A
Valve	"	)500	"	"	"	"	"	Every fifth	taken	"	"	"	"	"	"	"	Contract
Super-heater	At 100° F. 25-28	575	659	Ice	30-40	"	"	n)10%	"	"	"	"	"	Take at 212	"	"	Contract
No. 1 Lard	Not Taken	Not taken	Not taken	Plain Ice	35-40	"	Not taken	Not taken	"	taken	"	"	"	Not taken	"	"	18
Prime Lard	"	"	"	"	35-40	"	"	"	"	"	"	taken	"	"	"	"	18
Turbine	31-32	394	445	Ice & Salt	0-5	taken	"	"	"	Not taken	"	Not taken	"	70	"	"	Spec'ns

(- Not less than) - More than n) - Not more than W - Winter S - Summer

Methods for the Examination of Creosote Oil

To determine the specific gravity of the material simply heat the oil in a water bath until it is completely liquid. A glass stirring rod dipped into the liquid should show no solid particle on the rod when the same is withdrawn from the oil. When completely liquid stir thoroughly and fill the hydrometer cylinder, which has previously been warmed. Insert a specific gravity hydrometer of good make, taking care that the hydrometer does not touch the sides or bottom of the cylinder when the reading is taken. This reading should preferably be taken when the oil is at 15 degrees Centigrade. If difficulty is experienced in keeping the oil liquid at this temperature, the specific gravity may be determined at a higher temperature, preferably 38 degrees Centigrade and a correction made as follows: Multiply .0008 by the number of degrees Centigrade the oil is found to be above 15 degrees Centigrade and add the product to the observed gravity at the temperature at which the gravity is taken.

The distilling test is conducted as follows: The sample taken for analysis shall be strictly average of the whole bulk of oil to be tested. The oil shall be completely liquified and well mixed before any samples are taken. Whenever possible, a drip sample of not less than two gallons shall be taken, commencing after the oil has started to run freely. Where this cannot be done, as, for instance, in large storage tanks, samples shall be taken from various depths in the tank by means of a tube or bottle, the number of samples depending on local conditions.

For taking samples during the process of treatment a sample of the oil shall be taken from the storage tank about one foot from the bottom of the tank before the cylinder is filled, and when possible a sample directly from the cylinder during the process of treatment. For this purpose a thermometer well may be used.

The sample to be analyzed shall be thoroughly liquified by heating until no crystals adhere to a glass stirring rod, and also well shaken, after which one-half shall be taken for analysis on the balance reserved as a check test.

The apparatus for distilling the creosote oil shall consist of a glass stoppered retort, having a capacity as nearly as can be obtained of eight ounces up to the bend in the neck when the bottom of the retort and the mouth of the off-take are in the same plane. A nitrogen filled mercury thermometer of good standard make, divided into full degrees Centigrade, shall be used in connection therewith, placed with the bulb one-half (½) inch above the surface of the oil. In order to insure uniform results for comparative purposes, the length of the thermometer bulb shall be one-half inch; but in no case shall a thermometer with a long bulb be used. The bulb of the retort and at least two inches of the neck shall be and remain covered with a shield of heavy asbestos paper, during the entire process of distillation, so as to prevent heat radiation, and between the bottom of the retort and the flame of the lamp or burner two sheets of wire gauze, each 20-mesh fine, and at least 6 in. square, shall be placed.

The flame shall be protected against air currents. An ordinary tin can, from which a portion of the bottom and all of the top have been removed, placed on a support attached to the burner, will answer the purpose.

Before beginning the distillation the retort shall be carefully weighed and exactly one hundred grams of oil placed therein, the same being weighed in the retort. The thermometer shall be inserted in the retort with the lower end of the bulb one-half inch from the surface of the oil and the condensing tube attached to the retort by a tight cork joint. The distance between the bulb and the thermometer and the end of the condensing tube shall not be less than 20 nor more than 24 in. and during the progress of the distillation the thermometer shall remain in the position originally placed. The distillate shall be collected in weighed bottles and all fractions determined by weight on the basis of the dry weight of the oil. Reports shall be made on the following fractions:

0 to 170 degrees Centigrade.
170 to 200 degrees Centigrade.
200 to 210 degrees Centigrade.
210 to 235 degrees Centigrade.
235 to 270 degrees Centigrade.
270 to 315 degrees Centigrade
315 to 355 degrees Centigrade.
Residue above 355 degrees Centigrade.

Reports shall be made on individual fractions. In making such reports it is to be distinctly understood that these fractions do not necessarily refer to individual compounds. In other words, the fraction between 210 and 235 degrees will not necessarily be all naphthalene, but will probably contain a number of other compounds.

The distillation shall be continuous and at the rate of one or two drops per second.

When any measurable quantity of water is present in the oil, the distillation shall be stopped, the oil separated from the water and returned to the retort, when the distillation shall be recommenced and the previous readings discarded. In obtaining water-free oil it is desirable to free about 300 to 600 c. c. of the oil by using a large retort and using 100 grams of the water-free oil for the final distillation. In the final report as to fractions, a correction shall be made of the amount of water remaining, so that the report may be made on the basis of a dry oil.

This method with the diagram mentioned may be found on pages 430-431 and 432 of the Proceedings of the Twelfth Annual Convention of the American Railway Engineering and Maintenance of Way Association.

Specifications for Creosote Oil*

The oil must be pure coal-tar derivative of either English or German manufacture, preferably the latter.

The oil must have a specific gravity of 1.045, if German oil, and 1.030, if English oil, at 15 degrees C. It shall not contain over 3 per cent. permissable water unless an allowance of per cent. for per cent. is made by the seller if it contains over 3 per cent. and up to 6 per cent. of water; over 6 per cent. of water will not be accepted. The oil is to distil by the Von Schrenk method as follows:

Up to 210 degrees C., not over 5 per cent., all told, is to distil off.

Up to 235 degrees C., not over 25 per cent., all told, is to distil off.

The residue at the highest possible distillation (assumed to be about 360 degrees C.) should not exceed 4 per cent.; or if above 5 per cent. it must be soft.

An allowance of $.001 or fraction thereof is to be made by the buyer for any per cent. or fraction thereof beyond 6 per cent. that distils at 210 degrees C., and another allowance of $.001 to be made buyer for any per cent. or fraction thereof that distils beyond 25 per cent. to 235 degrees C. The quantity of the oil should be determined at 100 degrees F.

*Note. This specification was in use before the war. Since the war the conditions have changed, so that it is probable that the creosote used in this country will be of American manufacture. As a result the specifications will be somewhat different.

Method of Examining Petroleum Asphaltum

The sulphur is determined by the Eschka Method as described in the Report of the Committee on Coal Analysis published in the Journal of the American Chemical Society, Volume XXI No. 12, December, 1899. Care should be taken to heat carefully, to avoid loss of sulphur.

The method for taking the specific gravity is based upon the fact that a sample of the desired specific gravity will sink in .95 specific gravity alcohol and float on distilled water at 60 degrees Fahrenheit.

The method for taking the melting point of asphaltum pitches is the C. I. Robinson method, and the apparatus consists of a ring ½ inch deep and ⅝ inch diameter and a bicycle ball ⅜ inch diameter. The ring is ½ inch section cut from ⅝ inch brass pipe.

Directions for using: Press the ring full of the pitch cutting it off smooth with a hot knife. Place the ball in the center of the top surface of the pitch and suspend the apparatus in a beaker of water, the bottom of the ring being 1 inch from the bottom of the beaker; also suspend a thermometer in the beaker of water. Heat at the rate of 10 degrees per minute. The melting point is that temperature which the ball drops through the ring and reaches the bottom of the beaker. The pitch in the ring usually sags some time before the melting point is reached, but the temperature of the water at the time the ball reaches the bottom of the beaker is the temperature regarded as the melting point.

For test of 110 degrees Fahrenheit (43.3 degrees Centigrade) melting point pitch, the sample should be cooled to 50 degrees Fahrenheit (10 degrees Centigrade.) For 210 degrees Fahrenheit (98.8 degrees Centigrade) melting point pitch, cool to 80 degrees Fahrenheit (26.6 degrees Centigrade) or 100 degrees Fahrenheit (37.7 degrees Centigrade.)

The penetration test is made with the Dow Penetration machine according to the directions furnished with the machine.

The flashing point is determined by the method described in the Method of Determining Flashing and Burning Points of Combustible Liquids.

To determine the insoluble impurity, dissolve one gram of the sample in 50 to 75 c. c. carbon bisulphide and filter on a weighed Gooch crucible. Wash the beaker in which the sample was dissolved with carbon bisulphide several times. The increase in weight gives the amount of insoluble material in the sample.

Specifications for Petroleum Asphaltum of 180 Degrees

ADOPTED BY THE RAILWAY SIGNAL ASSOCIATION

1. The intention of these specifications is to provide an asphaltum suitable for placing around insulated wires in wooden trunking.

2. It shall be homogeneous and free from granular constituents.

3. It shall not contain over one per cent. of sulphur and shall be free from creosote, alkali, acid and water.

4. It shall be insulating, moisture-proof, and insoluble in water.

5. It shall have a specific gravity of between .950 and 1.000 at 60 degrees F. (15.5 degrees C.)

6. The melting point, as determined by the ball and ring method, shall not be under 170 degrees F. nor over 190 degrees F. (87.7 degrees C.)

7. The penetration determined by the Dow Penetration Machine for five seconds with 100 grams load on No. 2 needle shall be
   Not less than 3.5 m. m. at 77 degrees F.
   Not less than 1.0 m. m. at 32 degrees F.

8. It shall not flash below 300 degrees F. (149 degrees C.) in an open test cup.

9. It shall be soluble in pure carbon bisulphide to the extent of at least 98 per cent.

10. The manufacturer shall provide, at his refinery, all the apparatus and other facilities needed for making the required tests, and shall give the purchaser's representative an opportunity to test at all necessary times. Tests may also be made after delivery and the petroleum asphaltum will be rejected if it fails to meet the requirements of the specifications. The manufacturer shall pay freight both ways on all petroleum asphaltum rejected by the purchaser.

Method of Analysis of Wood Preservative Examination of Tar and Tar Acids

APPARATUS AND REAGENTS

The apparatus required by this method is a 2 c. c. pipette to measure the alcohol, and several 100 c. c. graduates provided with glass stoppers and feet, so that they will stand. The flat form is much better than the round form, since the solution of wood preservative in gasoline is moderately dark, and the readings can be much better made if the mass of liquid is in a layer thin enough, that light is readily transmitted through it.

Gasoline of the gravity specified is readily obtained in the market.

The caustic soda solution is made by dissolving a pound of commercial stick caustic soda in a quart of water, and diluting with water until it shows a gravity of 13 degrees Beaume. Of course, the gravity measurement should be made after the liquid has cooled to the temperature of the air.

OPERATION

Pour into a 100 c. c. graduate 80 c. c. of 88 degree Beaume gasoline, and add the material to be tested up to the 100 c. c. mark. Put in the stopper and shake thoroughly. Allow to settle until the tar settles to the bottom, or, as it happens in many cases, part of it settles and part adheres to the sides of the graduate. If the tar settles off nicely, none of it adhering to the sides of the graduate, read off the number of c. c. occupied by the tar, and calculate the amount as described below. If any of the tar adheres to the sides of the graduate, add a few c. c. of gasoline to a second of the same size, drain it, and then quickly but carefully pour the gasoline solution from the first graduate into the second, taking care that none of the tar goes along with the gasoline. Stopper the second graduate quickly, and read off the amount of gasoline solution in it. The difference between this reading and 100 c. c. when corrected for the error introduced by the volatility of the gasoline and the difficulty of pouring it all out, shows the volume occupied by the tar in the first graduate, from which the percentage can be calculated as described below.

To determine tar acids, pour 80 c. c. of the gasoline solution above referred to into a clean 100 c. c. graduate, provided the tar has been determined without transfer. If the transfer has been made, pour or suck out with a pipette the gasoline solution in the second graduate, until 80 c. c. are left. Then add 20 c. c. of caustic soda solution, and 4 c. c. ordinary alcohol. Shake thoroughly, and allow to stand until the material separates into two layers. The tar acids and part of the alcohol go into the lower layer. Read off the volume of this lower layer and calculate the amount of tar acids as directed below.

CALCULATION

(1) Tar, when it does not adhere to the graduate: Since 20 c. c. of the wood preservative are taken to start with, if volume of the tar obtained by reading the graduate is 1.5 c. c., for example, it is evident that the amount of tar is (1.5×100÷20) 7.5 per cent.

(2) Tar, when it adheres to the graduate: If the gasoline solution could be poured off completely from the tar into the second graduate, and if none of it volatilized, it is evident that the calculations could readily be made from the readings described. But experiments indicate that there is a little vaporization of the gasoline and little difficulty of pouring off completely. Accordingly, an allowance of 1 c. c. is made for these errors. Suppose, now, after the transfer, the reading of the second graduate is 97 c. c. This from 100 leaves 3 c. c. for the tar; but the error of the transfer is 1 c. c. Hence, the actual volume occupied by the tar in the first graduate is 2 c. c., and the amount is (2×100÷20) 10 per cent.

(3) Tar acids: Let us suppose that the reading of the graduate, after treatment with the soda and alcohol, as described, is 32 c. c. Twenty of these we added with the soda. Furthermore, experiment shows that 3 of the 4 c. c. of the alcohol added go into the lower layer with the tar acids and soda solution. Therefore, the increase in volume due to tar acids is (32-23) 9 c. c. Suppose, now, the volume of the tar found, as above described, is 2 c. c. Also, it is allowed that 1 c. c. of the gasoline solution is lost during the transfer from the first graduate to the second, whether this transfer is made after the tar determination or as a part of this determination, as described. There would be, therefore, 97 c. c. of the gasoline solution, which contains all the tar acids. But in the case suppose we find that 80 c. c. of this solution contain 9 c. c. of tar acids. Therefore, 97 c. c., or the whole of the gasoline solution, contains (80:97::9:10.91) 10.91 c. c. of tar acids, or this amount differently expressed is (10.91×100÷20) 54.55 per cent. This explanation may be briefly summarized as follows: Subtract 23 from the reading obtained when determining tar acids, and call this remainder a. Deduct from 100 c. c. the number of c. c. showing the tar and one more. Call this remainder b. Then make a proportion 80:b::a:x. x shows the volume in c. c. of the tar acids, and from this calculate the percentage.

NOTES AND PRECAUTIONS

The material to which this method applies is a distillate of Georgia pine, known under various trade names, such as Fernoline, Spirittine, pine oil, etc. It is, of course, not applicable to all wood preservatives, and those made from other kinds of wood and from coal tar do not behave, when treated as the method describes, like the Georgia pine distillate.

If the 20 c. c. of the distillate are poured into the graduate and then the gasoline added, it is much more difficult to get the gasoline and distillate thoroughly mixed than if the manipulation described is followed.

It will be observed that what is counted as tar is the material which separates from the distillate when treated with gasoline under the conditions described. No special effort has been made to determine what this is chemically, nor is it known whether the separation of the tar is complete or not. Also, there is some evidence that some of the gasoline remains dissolved in the tar, and increases its volume somewhat. No method is at present known of avoiding this difficulty, and accordingly the determination of the tar may be said to be in a sense arbitrary. But as long as the conditions and limitations of the method are known and understood, it is felt that no injustice by its possible and necessary errors is made.

There is quite a difference in distillates. Sometimes the tar will settle off nicely into a clean layer at the bottom of the graduate whose volume can be easily read off. More often, however, the tar separates in flocculent sticky particles or clots which adhere quite firmly to the glass. The cause of this difference in behavior is not known.

The object of adding a few c. c. of gasoline to the second graduate and draining before the transfer, is to compensate in the tar determination for the volume of the gasoline solution that adheres to the sides of the first graduate. While this source of error may not be serious compared with some necessary errors in the method, it is thought best to avoid known sources of error as much as possible.

It is perhaps hardly essential to mention that when the transfer is made to measure the tar, the two graduates used must be alike in measurement.

It will be observed that there is an error in the tar acid determination when the transfer is made as a part of the tar determination, due to the fact that some of the gasoline solution holding tar acids remains sticking to the sides of the first graduate. There is no means at present known of avoiding this error. It is believed, however, that this error does not amount to more than 1 per cent., and perhaps less.

The use of alcohol in the tar acid determination is due to the fact that without the alcohol the line between the two layers is not sharp. Apparently some of the soap formed by the combination of the tar acids with the soda does not dissolve in the soda solution in absence of alcohol. In its presence the difficulty wholly disappears.

Direct experiment, made by adding to a 100 c. c. graduate 200 c. c. of the soda solution, 80 of the gasoline, and 4 of alcohol, shows that 1 c. c. of the alcohol goes into the gasoline and 3 into the soda solution. It will be observed that it is assumed that the same thing takes place when the distillate is present.

The amount of gasoline vaporized during the transfer is, of course, affected somewhat by the temperature at which the transfer is made. Also the manipulation has an influence. It is believed that the 1 c. c. allowance should cover the necessary errors introduced by the volatility of the gasoline and the difficulties of the transfer.

The reason why so volatile a substance as gasoline is used to separate the tar is because no non-volatile material is known which separates the tar as satisfactorily as gasoline. Experiments with heavier non-volatile petroleum products have been made with unsatisfactory results. It is probable that further study will develop a more satisfactory solvent. It is, of course, well known that gasoline is not the only solvent that will separate tar from this distillate. It is, however, cheap and efficient, and may therefore reasonably be used until something better is suggested.

What is classed as tar acids, it will be observed, is what goes into the caustic soda solution. What these acids are, whether the separation of them from the neutral oils is complete, and whether anything else than tar acids and alcohol go into the soda solution, is not definitely known. In a sense, therefore, the determination of the tar acids is arbitrary.

Notwithstanding all the precautions that can be taken, it will be observed that the necessary errors of the method may be quite considerable. It is not expected that very good results can be obtained, but the limitations of the specifications under which the material is bought are so broad that very little difficulty has arisen, and the method proved an excellent means of keeping moderate control over an important commercial product whose chemistry is very little known.

DETERMINATION OF SPECIFIC GRAVITY

The test is made in the manner described in the Method of Determining Specific Gravity of Oils and Other Liquids.

COLD TEST

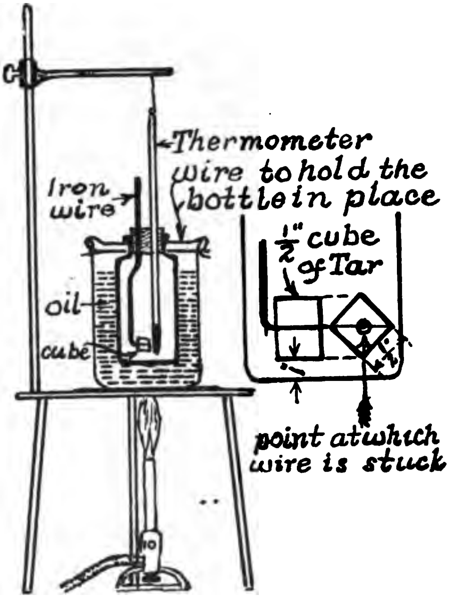
This test is made in the manner described in the Methods of Taking Cold Test and Chilling Points of Oils and Other Liquids.

FIRE TEST

This test is made in the manner described in the Methods of Determining Flashing and Burning Points of Combustible Liquids.

Cube Method for Determining Melting Point of Tars

Method for the Melting Point of Tars—Revised Methods adopted by the Special Committee on Bituminous Material for Road and Construction for American Society of Civil Engineers. Reference: Hubbard (Wiley and Son) Dust Preventative and Road Binders, page 381.


Thermometer
wire to hold the bottle in place
Iron wire
oil
cube
$\frac{1}{2}$" cube of Tar
point at which wire is stuck

The material whose melting point is to be determined is melted and poured into a mould that will make a one-half inch cube. A number 10 gauge wire about 6 to 8 inches long is bent at right angles for a length of ¾ inch at one end, and the center of the cube is placed on this end so that one of the diagonals of the vertical face of the cube is parallel to the long part of the wire.

Take a bottle of a size about 2 inches in diameter and 4 inches high, and place a piece of white paper in the bottom of it. Pass the long part of the wire through the cork of the bottle so that the lower edge of the cube will be within one inch of the bottom of the bottle. Also put a thermometer through the cork so that the bulb is opposite the cube.

Place bottle in a water or oil bath and raise temperature of the bath at the rate of 3 to 6 degrees Centigrade per minute. The melting point of the material is the temperature of the thermometer inside the bottle at the time that the material touches the paper in the bottom of the bottle.

NOTE—See Methods for Determining the Melting Points of Asphalts, by J. G. Miller and P. P. Sharpless; Seventeenth Annual Meeting, American Society for Testing Materials, June 20 to July 3, 1914

Analysis of Dead Oil of Coal Tar or Coal Tar Creosote

SAMPLE

About 1 quart of oil will be required for a complete analysis. The sample shall not be taken from the tank, can or other receptacle until the oil has been completely liquified and well mixed. One half shall be analyzed, as given below, and the other half held in reserve for a check test should such be desired.

SPECIFIC GRAVITY

The specific gravity of the oil shall be obtained by nearly filling the hydrometer cylinder with the liquified oil and placing the cylinder in H_2O, which shall then be heated until the temperature of the oil is several degrees higher than that at which the specific gravity is to be taken. The H_2O is then allowed to cool until the oil has reached a temperature of 38° C. The oil is then thoroughly stirred, the hydrometer placed in the cylinder, and the reading carefully taken. Care must be taken that the hydrometer does not touch the bottom or sides of the cylinder, when reading.

FLUIDITY

The fluidity of the oil shall be determined by heating about 100 c. c. of the oil in the beaker, to a temperature of not less than 45° C. and then allowing the oil to cool gradually. When a temperature of 38° C. is reached the oil shall be carefully examined, using a glass stirring rod. No solid crystalline particles should be present on the rod when withdrawn from the oil.

DISTILLATION

The apparatus for the distillation of the oil shall be arranged as shown below. Before attaching the retort to the ring stand it should be accurately weighed on the pan balance. Then without removing the retort from the balance, pour exactly 100 grams of the thoroughly liquified oil into the retort. This can readily be done by using a glass dropper to add the last drops.

ARRANGEMENT OF DISTILLATION APPARATUS

Before distillation is begun, the thermometer must be so fitted through the stopper in the neck of the retort that the bulb is 1/8″ above the surface of the oil, and remain thus until the operation is complete.

The heat applied to the retort must be so regulated that a complete distillation will require from 35 to 40 minutes. The naphthalene fraction shall be collected in from 10 to 12 minutes.

It is to be understood that the distillation has begun as soon as the first drop of the distillate appears in the delivery tube of the retort, and ended as soon as a temperature of 300° C. has been reached.

The distillation of some samples of oil may be accompanied, especially during the first few moments, by a violent spattering of the liquid, and small quantities may go over into the test tubes. This can usually be avoided by gently shaking or moving the ring stand and retort so that the oil is kept in motion.

DISTILLATE

The fractions of the distillate are separated at the temperature given in the table below and collected in test tubes. The weights of the test tubes shall be accurately determined before the distillation. After the completion of the distillation, the test tubes and the retort with their contents shall be accurately weighed and the per cent. of the various fractions computed.

Test Tube	Temperature	Fraction
No. 1	0°-170° C.	Water, hydrocarbons and phenols.
No. 2	170°-205° C.	Phenol and cresols.
No. 3	205°-210° C.	Naphthalene and phenols.
No. 4	210°-235° C.	Naphthalene.
No. 5	235°-240° C.	Naphthalene and anthracene oil.
No. 6	240°-270° C.	Anthracene oil.
No. 7	270°-300° C.	Anthracene oil and anthracene.
No. 8	300° and above	Residue in retort.

The per cent in fractions Nos. 3 and 4 shall be added together for the determination of the naphthalene and should give 40% or more. The per cent. of Nos. 7 and 8 shall likewise be added together and their sum must not be less than 20%.

The sum of the percentage in all the fractions should be ascertained and the difference from 100% recorded as loss per cent. The loss should not exceed ½ of 1% (.5%) except when there is a considerable amount of water present. If the loss is found to be large when a sample containing little water or other low boiling constituents is distilled, the distillation should be repeated after the computations have been repeated in order to insure their freedom from error.

WATER

To determine the amount of H_2O in the oil, transfer the contents of the test tube No. 1 to a small separatory funnel. If the separation of the water from the other constituents of the funnel is well defined, draw off the water in a test tube and compute its weight. If the separation be not well defined, pour a small amount of ether in the funnel and shake. Allow the contents to settle and draw off the water from the ethercal solution. Weigh the water and compute the per cent.

The ethercal solution shall then be warmed, to expel the ether, and the oil or remainder reserved for the next test.

The ether used should be free from alcohol and may be prepared in the following manner: Shake a convenient quantity of sulphuric ether in a separatory funnel with one-half its volume of distilled water and then let the water run out. Repeat the washing of the ether twice with distilled water (fresh lots).

TAR ACIDS

The volume of the tar acids in the oil shall be determined in the following way: The contents of all the test tubes shall be transferred to a 300 c. c. beaker and mixed thoroughly by stirring. 30 c. c. of caustic soda solution (Specific Gravity 1.20) shall be added to the oil and the whole gently heated for 2 minutes with constant stirring. The oils and soda solution shall then be transferred to a large separatory funnel and shaken vigorously for at least 1 minute. The mixture shall then be allowed to settle and the soda solution, which forms the lower layer drawn off and saved. This operation must be repeated a second and third time using 20 c. c. of the soda solution.

The three soda solutions shall now be mixed together, boiled vigorously for 5 minutes and allowed to cool. Dilute sulphuric acid (1:3) is then added until the solution begins to change the litmus from blue to red. About 35 c. c. H_2SO_4 will be required and care must be taken to keep the solution cool while adding the acid.

The entire solution is now transferred to a separatory funnel and allowed to stand until the tar acids are well separated. The volume of the tar acids shall then be measured in a graduate.

ACETIC ACID OR ACETATES

In order to determine whether the oil contains acetic acid or acetates, the following method shall be employed:

80 grams of oil, thoroughly liquified, shall be placed in a retort and 6 c. c. H_2O plus 1 gram of chemically pure H_3PO_4 is added. Distill the mixture rapidly up to 170° C. and collect the distillate in a test tube as before. If the spattering cannot be prevented in the usual manner and some of the oil is carried over into the test tube, another distillation must be made of the test tube contents in a distilling flask. Transfer in the distillate to a small separatory funnel, separate and draw off the water. Add 5 c. c. of standard alkali litmus solution to the water distillate. Should the latter

cause the litmus to turn red, acetic acid or acetates are present in the oil.

REPORT OF ANALYSIS

The reports of the analysis of the oil should be recorded in the following form:

ANALYSIS OF DEAD OIL OF COAL TAR

Sample No.:
Manufactured by:
Purchased from:

SUMMARY OF RESULTS

Specific Gravity at 38° C.:
Condition at 38° C.:

Fractionation.

Weight of retort. Weight of retort and oil.

Fraction No.	Temperature	Pec cent.	Wt. of vessel	Wt. of vessel and contents	Wt. of contents
1	° -170° C.				
2	170-205° C.				
3	205-210° C.				
4	210-235° C.				
5	235-240° C.				
6	240-270° C.				
7	270-300° C.				
8	Residue °C.				

Total per cent. found: 99.50
Loss per cent. - - - - .50

Naphthalene:	Per cent.
Oil not distilling below 270 C.:	Per cent.
Water:	Per cent.
Tar Acids:	Cubic centimeter.
Acetic Acid and Acetates:	

CLEANING APPARATUS

The retort and test tubes should be thoroughly cleaned after each operation. To remove the residue from the retort add some oil and heat until residue is all dissolved. Pour out the oil and wash with hot water and sapolio.

Note A: The standard alkaline solution of litmus is prepared by dissolving powdered litmus in 250 c. c. of H_2O until a deep blue color is developed. 1 gram of C. P. NaOH is added and the whole diluted to 500 c. c. with distilled water.

PART IV

NOTE: The following methods of analysis in Methods Nos. 68 to 81, inclusive, are special methods used by the Pennsylvana Railroad in examining special paints bought under specification. The analyses were made to see how closely the paint checked with the specifications. If anything peculiar was noticed about the paint a thorough analysis was made, and the methods of this thorough analysis varied in different cases, depending upon conditions. These methods are given as special methods strictly applicable to these paints only, but similar paints could be analyzed by similar methods.

Method for the Analysis of Freight Car Color

DETERMINATION OF OIL, PIGMENT AND MOISTURE, OR DEFICIENCY OF HYDRATION

EXPLANATORY

The standard freight car color of the Pennsylvania Railroad Company is bought in paste form, and the paste must contain nothing but oil, pigment and moisture. The moisture and other volatile constituents must not exceed 2 per cent. of the weight of the paste, and the paste must not be a "liver" when received. The oil must be pure raw linseed oil, and must not be less than 23 nor more than 27 per cent. of the weight of the paste. In determining the proportions of oil, pigment and moisture, or other volatile constituents, the oil must be heated before weighing to 250 degrees Fahrenheit, and the pigment must be dried before weighing at from 60 to 90 degrees Fahrenheit in air which has been artificially deprived of moisture. The inert material in the pigment may be sulphate of calcium or gypsum, silica, kaolin, soapstone or asbestos, the two former preferred. Carbonate of Calcium must be present to the extent of 2 per cent. but must not exceed 5 per cent. of the weight of the pigment. Ground feldspar is not desired, and barytes or sulphate of barium, organic coloring matters and caustic substances are excluded. The pigment must contain not less than 20 per cent. of sesquioxide of iron, and if sulphate of calcium or gypsum is present, it must be fully hydrated. The paste must conform to standard shade, and must pass test for fine grinding.

APPARATUS AND REAGENTS

The flasks and beakers required are perhaps sufficiently designated under "Operation."

The arrangement for taking moisture out of the air used in drying the pigment by causing it to bubble through concentrated oil

of vitriol may, perhaps, be readily improved in every laboratory. Derschsel's wash bottles for washing gas, with ground glass joint, are very convenient for this purpose.

The gasoline specified is readily obtained in the market. It is best to obtain it in tin cans and every new shipment should be tested. If the same amount used in an analysis leaves weighable residue, when a blank oil determination is made, a correction corresponding to this should, of course, be made. It is better, however, to secure such a grade of gasoline that no residue will be left. If the gasoline is not shipped or stored in wood or dirty cans, very little difficulty will occur.

The Ethyl alcohol is the ordinary 95 per cent. alcohol on the market, and the other mentioned later is the ordinary commercial sulphuric ether of the United States Pharmacopia.

OPERATION

Weigh a six-ounce Erlemeyer flask and then introduce five grams of the paste to be examined. The manipulation of the paste is not entirely easy. It is best to weigh the material into a flask, using a narrow spatula to transfer it and taking great pains to prevent any of the paste from getting on the outside of the flask, or near the top of the inside. Fill the flask about one-third full with 88 degree Beaume gravity gasoline, and agitate with a rotary motion in a horizontal plane, until the paste is all decomposed. Now add more gasoline and agitate in the same way to secure mixing, until the flask is about two-thirds full, and finally add gasoline from the jet of a wash bottle, so as to mix as thoroughly as possible, until the flask is nearly full. Cork loosely, without permitting the liquid to touch the cork, and allow to settle, which may require from two hours to two days. When the liquid is clear, carefully remove the cork, and decant the liquid into a tall lipless beaker, holding about nine ounces. By using sufficient care, the liquid may be decanted down so that not over five cubic centimeters are left in the flask. Some skill and a little experience are required to secure this result. Incline the flask and allow perhaps half the liquid to run out. Then if the pigment has not already collected at the lowermost point of the flask, keep the flask inclined just so the liquid will not run out, and assist the collection of the pigment at the lowermost point, by striking the flask gently against the desk. If this operation roiles the liquid near the bottom of the flask, place it still inclined in the top of the beaker or other support and allow to settle again, which usually takes only a short time. Then continue the decantation until the limit is reached. Place the beaker where the temperature is a little above the boiling point of the liquid, and where there are no naked lights and then fill the flask with gasoline again in the manner before described. Allow to settle a second time, and repeat the decantation in the same manner. Enough of the liquid in the beaker will, if the evaporation is properly managed, go off while the pigment is settling the second time to furnish room for the liquid for the second decantation. Evaporate the liquid in the beaker as before, gradually raising the temperature as the liquid will bear it, until a temperature of 250 degrees Fahrenheit is reached. Cool and weigh from time to time, and continue the heating at the same temperature until constant weight is obtained. This weight, minus the weight of the beaker, is the weight of the oil. After the second decantation add to the flask containing the pigment three or four cubic centimeters of a mixture of equal parts of Ethyl alcohol and distilled water, agitate to secure thorough mixing, cork with a double perforated rubber stopper carrying two tubes, one of which reached to within an inch of the bottom, and attach the other to a steam or water aspirator, or other means of drawing air through the flask. The air drawn into the flask should not carry dirt, or injurious gases along with it. The gasoline, the alcohol and the principal portion of added water are removed in the course of a few hours. As soon as the visible liquid has disappeared, attach to the air inlet tube an arrangement for passing the air through concentrated oil of vitrol, and continue the drying until the flask containing the pigments show constant weight. Deduct the weight of the flask from this weight, and proceed as explained under "Calculations."

CALCULATIONS

The weight obtained by deducting the weight of the beaker from the constant weight of the beaker and oil as above described, gives the weight of oil in 5 grams of the paste. Let us suppose this to be 1.1865 grams. Then the percentage of oil would be (1.1865×100÷5) 23.73. Also if the pigment in the paste were fully hydrated, the weight of the flask and pigment, minus the weight of the flask, gives the weight of the pigment in five grams of the paste. Suppose this to be 3.7240 grams. Then the percentage of pigment would be (3.7240×100÷5) 74.48. In this case, if no volatile constituents but moisture is present, the moisture would be 100—(23.73 plus 74.48) 1.79 per cent., the moisture being determined, as in one, by difference. In case the pigment in the paste was not fully hydrated, the water added with the alcohol accomplished this result, and the sum of the oil and pigment, provided no other volatile constituent was present, always exceed 100 per cent., the excess representing the dificiency of hydration of the pigment as is readily seen.

NOTES AND PRECAUTIONS.

It is quite apparent that this method involves as its principal features the insolubility of the pigment in gasoline, the solubility of the oil in the same menstruum, and the volatility of the gasoline without vaporizing either the oil or the pigment.

It frequently happens that samples of paste are found, the pigment of which settles very slowly. With many of these samples, the addition of 3 to 5 cubic centimeters of mixture of equal parts of ethyl alcohol and distilled water, while decomposing the paste, facilitates the settling.

It is best to add this alcohol and water before the second addition of gasoline in order to secure thorough mixing. After a little experience is gained, the behavior of the pigment when the paste is decomposed and before the second addition of gasoline is something of a guide as to whether the alcohol and water are needed. If the pigment shows a disposition to settle off readily on allowing the flask to stand a few minutes, the alcohol and water will probably not be needed. If there is no such disposition, it is better to add them. No harm results from this addition, and some operators prefer to always add the alcohol and water. With a very obstinate paste which settles very slowly, or indeed refuses to settle clear after considerable time, it is usually best to start afresh and use a mixture of equal parts benzol and alcohol in place of the first addition of gasoline, and sometimes ether may be used to advantage throughout.

There is considerable evidence that the rapid settling of the pigment is a question of the hydration. During the grinding the mills usually become quite warm, and the tendency is to dehydrate both the sulphate of calcium and the clay, both of which are almost universally present in greater or less amount in freight car color. Alcohol containing small amounts of water is slightly soluble in gasoline, and its presence, therefore, facilitates the transfer of the water to the pigment. Also commercial ether contains small amounts of water, and this is apparently transferred to the pigment in the same manner. With some points a coagulation of the pigment in flakes, and almost immediate tendency to settle, follow the addition of the alcohol and water, or the use of the ether.

It should be stated that, notwithstanding all precautions, it sometimes happens that some extremely fine portions of the pigment refuse to settle even after a day or two, leaving a slight tint or opalescence in the liquid. No method is known of overcoming this difficulty, but it is believed that the error resulting does not exceed a small fraction of one per cent.

If the paste contains 25 per cent. of oil, as is desired and expected, the amount of oil in five grams would be 1.2500 grams. About 150 cubic centimeters of liquid is present before the first decantation, and by the supposition five one-hundred and fiftieths of this are left after the decantation is finished. That is, (1.2500×5÷150) 0.0416 gram of oil are left. But if the directions are followed, five one-hundredth and fiftieths of this are left after the second decantation, that is (0.0416×5÷150) 0.0013 gram of oil are left with the pigment and weighed with it. This amounts to an error of (0.0013×100÷5) 0.026 per cent. If greater accuracy than this is desired, a third treatment with gasoline can be employed.

The separation of the liquid from the pigment by decantation is much better than to use a siphon. Formerly a siphon was employed, but it was found that there was a little loss due to material adhering to the siphon, and also the liquid could not be drawn off so as to leave as small a volume behind, on account of the currents at the inlet and disturbing the pigment.

It is probable that there is a slight oxidation of the oil during the evaporation and subsequent drying to constant weight. Direct experiments on oil free from moisture, however, show that the changes in weight due to this oxidation is very small. Mulder has shown that during exposure to the air, especially at high temperatures, linseed oil loses carbon and possibly hydrogen while it gains oxygen, and experiments made for the purpose show that the loss and gain very nearly balances each other, so that the error introduced during the drying can safely be ignored.

The directions require that both the oil and pigment be dried until constant weight is obtained. It is probable that, especially with the oil, absolute constant weight would never be obtained. If the difference between two weighings an hour apart does not exceed one or possibly two milligrams, the resulting error will be small, as is readily seen, as to have no practical importance.

The directions to put the paste low down in the flask during the weighing and to prevent the liquid from touching the cork, are perhaps of more importance than would appear at first sight. The difficulty of avoiding loss while decomposing the paste, if it is near the top of the flask, is quite considerable, and the loss if the liquid touches the cork is much more than would be supposed.

Gasoline is quite sensitive to changes of temperature, and its vapor tension even at ordinary temperature is quite considerable. If the flask is tightly corked, therefore, there is danger of loss of both flask and its contents. If the pigment is fully hydrated, the amount of pigment, liquid and moisture or other volatile constituents are given by the method with all the accuracy that is necessary in the analysis of such a product as freight car color. But if, as frequently happens, the pigment is not hydrated, it is evident that benzine, turpentine or some other volatile substance could be added equal in amount to the lack of hydration of pigment, without this fact being revealed by the method as described. In cases where such additions are suspected, their presence or absence is determined by other tests, especially by distilling over the volatile constituents from a portion of the paste and examination of the distillate.

DETERMINATION OF SESQUIOXIDE OF IRON

The pigment of the standard freight car color is a sesquioxide of iron, with a little carbonate of calcium, and quite a large amount of various inert material as extenders or fillers. These may include kaolin, gypsum, ground soapstone, silex, feldspar and asbestine or ground asbestos, as well as the natural impurities occurring in the iron ores, frequently used to furnish the required sesquioxide of iron,—barytes, or sulphate of barium excluded. A minimum percentage of sesquioxide of iron being an essential feature of the specifications, a rapid and at the same time accurate method for its determination is desirable. The directions given below seem to secure this result.

APPARATUS AND REAGENTS

The apparatus required by this method is simply the beaker, burettes, pipettes, etc., present in every laboratory, and requires no special comment.

The Stannous Chloride Solution is made by adding to one pound of concentrated chemically pure hydrochloric acid one pound of chemically pure stannous chloride obtained in the market. Dissolve by stirring. If all the stannous chloride fails to dissolve after a little time, add enough water to secure solution. Dilute to two liters. The bottle holding this solution should be kept closed when not in use, and a few fragments of metallic tin added to keep the salt reduced to the stannous form.

The Permanganate of Potash Solution for titration is made as follows: To one liter of water add two grams of crystallized permanganate of potash, and allow to stand in the dark not less than a week before using. Determine the value of this solution in terms of metallic iron. For this purpose 150 to 200 milligrams of iron wire or mild steel are dissolved in 15 cubic centimeters of chemically pure hydrochloric acid (1.10 specific gravity). After solution is complete, reduce with a stannous chloride, add mercuric chloride, pour into solution of phosphoric acid, and titrate exactly as described below. It is, of course, essential that the amount of iron in the wire, or soft steel, should be known. The standard in use in the Pennsylvania Railroad Laboratory is a mild steel in which the iron is known by determining carbon, phosphorus, silicon, sulphur, manganese and copper, and deducting the sum of these from 100 per cent. Not less than two independent determinations should be made, and three are better. The figures showing the value of the permanganate solution in terms of metallic iron should agree to a hundredth of a milligram in the different determinations. A very satisfactory method of making and keeping permanganate of potash solution is as follows: Have a large glass bottle holding, say eight liters, and two of half the size. Paint the outside of these bottles with several coats of black paint or varnish. Fill the large bottle with the standard solution, and after it has stood a proper time fill one of the smaller bottles from it without shaking and standardize. At the same time fill the second small bottle, and refill the large one. When the first small bottle is exhausted, standardize the second small one and fill the first from stock. When this is exhausted, standardize the first again and fill the second from stock, refilling the stock bottle and so on. By this means a constant supply of sufficiently matured permanganate is always available. Of course, if the consumption is very large, larger bottles or more of them may be required. Since changes of temperature affect the volume of all solutions, it is desirable that the permanganate solution should be used at the same temperature at which it was standardized. With the strength of solutions recommended, if the permanganate is used at a temperature of 20 degrees Fahrenheit, different from that at which it was standardized, the error amounts to less than 0.05 per cent. in a pigment containing 80 per cent. of sesquioxide of iron.

The Mercuric Chloride Solution is made by adding to a bottle containing about a pint of distilled water two ounces of chemically pure mercuric chloride, filtering if necessary. A saturated solution is desired for use and the above amount of mercuric chloride is sufficiently in excess, so that the water can be added from time to time.

The Phosphoric Acid Solution is made as follows: Add 160 grams of chemically pure manganese sulphate, obtained in the market, to sufficient water, and when solution is complete, dilute to 1750 cubic centimeters. Then add 330 cubic centimeters of syrupy phosphoric acid, (1.70 specific gravity) obtained in the market, and 320 cubic centimeters of concentrated pure sulphuric acid. Mix thoroughly, filter if necessary, and keep it in a well-stoppered bottle.

OPERATION.

Remove the pigment from the six-ounce Erlemeyer flask, in which it is left in the methods describing how to separate the pigment from the other constituents of freight and passenger car colors, by means of a spatula, taking care to get out all that can be so obtained and detected. The pigment, being received on smooth or glazed paper is then mixed with the spatula, the lumps which are apt to be present being crushed in the operation. A uniform sample being obtained, weigh into a small porcelain crucible half a gram of the pigment, and ignite to destroy organic matter, avoiding very high temperatures. Put the ignited pigment into a two or three ounce beaker, and dissolve in hydrochloride acid, taking pains to clean the crucible by washing with some of the acid, and rubbing with a rubber tube on the end of a glass rod. Use in all 15 cubic centimeters of chemically pure hydrochloric acid (specific gravity 1.19). In dissolving use heat; solution usually takes only a few minutes. In cases of a refractory pigment, a few drops of the standard stannous chloride solution added from time to time greatly hastens solution; an excess should be avoided. When solution is complete, if stannous chloride has not been used, the solution will, of course, have the reddish color characteristic of ferric chloride. If stannous chloride has been used, even in very slight excess, the solution will be nearly colorless. The subsequent manipulation in the two cases differ. In the first case, while the liquid is still hot, add from a burette or pipette the standard stannous chloride drop by drop with agitation until the solution has lost the greenish yellow color, which is characteristic of the partially reduced solution. In case stannous chloride has been used to secure solution, add permanganate of potash solution, until the greenish yellow color characteristic of a partially reduced iron solution appears, and then add stannous chloride from a burette or pipette, until reduction is complete, as above described. Dilute with about 200 c. c. water. Add now to the reduced solution 5 c. c. of mercuric chloride solution, taking care to wash down the sides of the beaker containing the reduced solution with the mercuric chloride. Mix thoroughly and then pour the reduced solution into a beaker holding 500 c. c. and containing 200 c. c. of water, to which has been added 10 c. c. of phosphoric acid solution. Titrate now with standard permanganate, adding with moderate rapidity at first, then more slowly with constant stirring, until one drop gives the pink color characteristic of the end reaction.

CALCULATIONS

An example of all the calculations is given herewith. The soft steel employed in standardizing permanganate of potash solution in the Pennsylvania Railroad Laboratory contains 99.27 per cent. metallic iron. 0.1498 grams of this contains, therefore, (.1498×.9927) .1487064 grams of metallic iron. This required 42.99 cubic centimeters permanganate solution, or 1 cubic centimeter of permanganate solution is equal to (.1487064÷42.9) .003466 metallic iron. But metallic iron is to sesquioxide as 112 to 160 (Fe_2 to Fe_2O_3). Hence 1 cubic centimeter of permanganate is equal to (112 : 160 : : .003466 : ×) .004951 sesquioxide of iron. Twice the number of cubic centimeters used in the analysis, since half a gram is taken, multiplied by this factor, gives the sesquioxide of iron in one gram of the pigment, and this multiplied by 100 gives the percentage.

NOTES AND PRECAUTIONS.

It will be observed that this method is almost exactly the Zimmerman-Reihardt method described by Mixer and Dubois in the Journal of the American Chemical Society for May, 1895. In our hands the method has given such admirable results that we use it wherever possible.

The pigment frequently adheres with considerable tenacity to the sides and bottom of the flask used in separating it from the other constituents of the paint. Moreover, the constituents of the pigment separate from each other in the gasoline solution, the heavier particles being the first at the bottom. It would lead to error, therefore, to take sufficient material for an analysis out of the flask without previous care to secure a uniform sample. The flask should be well cleaned, and the whole amount thoroughly mixed before weight is made.

Pigment separated from oil by means of any solvent is apt to contain some organic matter, possibly oxidized oil, or trace of the unoxidized oil, not removed. Moreover, the passenger car color pigment contains as an essential constituent organic coloring matter. These substances interfere with the subsequent titration with permanganate, and must accordingly be removed. This is apparently easiest done by ignition as described. If very high temperatures are used, the subsequent solution will be more difficult.

The use of stannous chloride to facilitate solution and also to reduce the iron requires some precaution. The more stannous chloride there is present before the mercuric chloride is added, the more of this latter reagent must be added to decompose it, since it is impossible to titrate in presence of stannous chloride. But the more mercuric chloride added to decompose stannous chloride, the more mercurous chloride there is present in the solution to be titrated. Experience shows that this mercurous chloride may lead to erroneous results in the titration. If the material to be titrated is opalescent only from suspended mercurous chloride, entirely satisfactory results may be expected. If, on the other hand, a voluminous precipitate of mercurous chloride is present, the results may be seriously in error, possibly due to a reaction between the mercurous chloride and the permanganate.

The use of the phosphoric acid solution enables the titration with permanganate to be made in presence of hydrochloric acid, and thus avoids displacing this acid by sulphuric, and the use of the reductor, which are characteristic of the older methods. Apparently the manganese sulphate is the important factor in this possibility, the phosphoric acid being added to give a colorless solution, so that the end of the reaction may be observed with sufficient sharpness.

It will not escape notice that if the manganese sulphate, the phosphoric acid, or the sulphuric acid, used in making the phosphoric acid solution, any or all of them contain iron in the protoxide form, or even organic matter, an error may be introduced in the titration. It is well, therefore, to test this solution with permanganate before using it. If the same amount that is required for an analysis uses up more than one, or possibly two drops of permanganate, the amount actually used up must be deducted from the figure obtained in the regular titration.

DETERMINATION OF BARIUM SULPHATE

The presence of barium sulphate can be detected in the hydrochloric acid solution for the determination of sesquioxide of iron, by the settling out of the very dense and heavy precipitate of insoluble barium sulphate. Swirl the beaker, and if barium sulphate be present it will very readily come to the bottom; much quicker than any silica or other substances which might be present. If found, filter off residue, wash with hot 10 per cent. hydrochloric acid and cold water several times. Ignite residue. Now fuse ignition with 4 or 5 times its bulk of a mixture of equal parts sodium carbonate and potassium carbonate. Fuse 15 minutes, cool and put crucible and contents into a 250 c. c. beaker half full of water. Boil to therough disintegration and solution of soluble portions. Filter and wash well with hot water. Filtrate contains the sulphate and the residue on the filter the barium in the form of barium carbonate. Dissolve residue in 5 c. c. hydrochloric acid and 95 c. c. water. Add about 30 c. c. calcium sulphate (saturated solution) and 2 or 3 drops ammonium hydroxide, but keep solution distinctly acid. Boil 10 or 15 minutes, let precipitate settle, filter, wash and ignite. Note that no barium sulphate passes through filter paper. Weigh as barium sulphate.

To the filtrate consisting probably of sodium sulphate, add hydrochloric acid until faintly acid, boil and add 15 c. c. of a 10 per cent. solution of barium chloride. Boil 15 minutes, let precipitate settle, filter, ignite, and weigh as barium sulphate. The latter should check the former.

DETERMINATION OF CALCIUM CARBONATE

Weigh one gram of the dry, well mixed pigment into a 6-ounce, wide mouth Erlemeyer flask, for the determination of carbon dioxide. Close the stop-cock of the dropping funnel and fill the bulb about two-thirds full of ¼ sulphuric acid. Place stopper in position, open funnel stop-cock, at the same time opening suction line to give gentle suction so that acid will run slowly into flask. Regulate the suction so that the potassium hydroxide solution in the Geissler bulb will bubble at the rate of about three bubbles per second. The potassium hydroxide solution should be made to have a specific gravity of 1.27. Heat the solution in the flask to boiling and keep at incipient boiling for about 15 minutes. Aspirate 20 minutes. Disconnect the Geissler bulb, and the calcium chloride tube, and weigh. Before starting a determination, always get a blank weight of the Geissler bulb and calcium chloride tube by aspirating for twenty minutes and weighing. Then replace, aspirate twenty minutes more and weigh again. A constant weight should be obtained, which is used as a blank.

CALCULATION

Molecular weights of calcium carbonate is 100.1; molecular weight of carbon dioxide is 44.0. Therefore, weight of carbon dioxide obtained is the weight of calcium carbonate as 44.0 is to 100.1, or, dividing weight of carbon dioxide obtained by 0.44 and multiplying this result by 100 will give the percentage of calcium carbonate sufficiently accurate. This specification assumes that all the carbon dioxide is present as calcium carbonate.

DETECTION OF CALCIUM SULPHATE

To a small portion of pigment in a beaker, add cold water. Stir well and filter, and to the filtrate add a few drops of a saturated solution of ammonium oxalate, with a few drops of ammonia. If any calcium sulphate is present, the characteristic percipitate of calcium oxalate will be obtained.

DETERMINATION OF FINENESS

The pigment of the paste must be so fine that after having been separated from the oil and freed from hygroscopic moisture, and then thoroughly mixed again with pure raw linseed oil, which has also been freed from moisture, in the proportions of one part oil to one part pigment by weight, it will stand the following test, viz: Place a small amount of the above mixture on one end of a strip of dry glass, set the strip vertical where the temperature is maintained at 70 degrees Fahrenheit, and allow it to remain undisturbed for half an hour. The mixture runs down the glass in a narrow stream, and if the pigment is fine enough, the oil and pigment does not separate for at least an inch down from the top of the test.

DETERMINATION OF SHADE

The shade of paint being affected by the grinding, the P. R. R. standard shade is that given by the dry sample sent, mixed with the proper amount of oil and ground, or better rubbed up in a small mortar with pestle until the paste will pass P. R. R. test for fineness. It is best to use samples of the dry pigment for each day's testing. The comparison should always be made with the fresh material and never with the paint after it has become dry. The comparison is easiest made by putting a small hillock of the standard paste and of that to be compared near each other on glass, and then laying a thin piece of glass as cover on the two hillocks and pressing them together until the two samples unite. The lines where the two samples unite is clearly marked, if they are not the same shade. It is recommended to use for the cover the thin glass commonly employed by microscopists, since this thin glass does not change the shade perceptibly.

MAUMENE'S TEST FOR PURITY OF OIL

Fill a 1000 c. c. copper beaker about one-fourth full of the paste under examination, and then fill nearly full with 85 degrees Beaume gasoline. Mix thoroughly with spatula, add about 25 c. c. denatured alcohol, cover with watch glass and allow to stand until the pigment has settled and the supernatant liquid is perfectly clear. It will take about four hours for the pigment to settle, and it is better to allow to stand over night. When settled, decant, or pipette off the supernatant liquid, in which the oil is dissolved, into a tin cup and heat on hot plate at a temperature of 250 degrees Fahrenheit for four hours. Cool and pipette 5 c. c. into a dry, 100 c. c. beaker which must not weigh less than 19 grams and not more than 20 grams. Add 10 c. c. standard sulphuric acid, stirring with a thermometer having a small bulb, and noting the temperature at frequent intervals.

Record the highest temperature reached and maintained for an appreciable length of time. The temperature of the oil and standard sulphuric acid should be the same before mixing. A good linseed oil should show a temperature rise of at least 100 degrees Fahrenheit. The standard sulphuric acid should be of such strength that 100 c. c. will raise 30 c. c. water 100 degrees Fahrenheit when placed in a 100 c. c. beaker weighing not less than 19 grams and not more than 20 grams. By adding 15 c. c. water to a liter of commercial sulphuric acid about the proper strength will be obtained.

SEE NOTE—PART IV.

Method of Analysis of Building Paints

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF FINENESS AND SHADE

These determinations are conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF LEAD OXIDE

Weigh .5 gram of pigment (which has been well ground and mixed in a mortar) into a 250 c. c. beaker, add a mixture of 5 c. c. water, 5 c. c. ammonium hydroxide (Sp. Gr. .90) and 15 c. c. of 50 per cent. acetic acid. Cover with a watch glass and place on steam plate for one-half hour. Remove from plate, boil vigorously on gauze for about one minute and filter into a 250 c. c. beaker, washing thoroughly with a 2 per cent. solution of ammonium acetate. To the filtrate add 50 c. c. concentrated nitric acid, 12 c. c. concentrated hydrochloric acid and 1 c. c. concentrated sulphuric acid. Cover with a watch glass on glass triangle and evaporate to syrup on steam plate. Lead and zinc are now present as sulphates. Add 3 to 4 drops concentrated sulphuric acid and about 25 c. c. water, heat on steam plate one-half hour. After washing sides of beaker with distilled water, filter off the insoluble lead sulphate, washing well with a 2 per cent. solution of sulphuric acid but using not more than 160 c. c. Save filtrate for determination of zinc oxide. Transfer filter paper and residue to original beaker, add 10 c. c. ammonium hydroxide (Sp. Gr. .90) and 25 c. c. of 50 per cent. acetic acid. Heat to complete solution. Filter into a 1000 c. c. beaker, washing thoroughly with hot water. Fill beaker to about 700 c. c. with water, bring to boil and add 15 c. c. of potassium bichromate solution (36 grams potassium bichromate per liter of water). Boil gently for about five minutes. Allow precipitate to settle and filter through a weighed Gooch crucible. Dry residue on gauze but do not ignite strongly. Cool and weigh as lead chromate. The lead should be reported as per cent. of lead oxide.

Weight of lead chromate times the factor .6906 times 2, times 100, equals per cent. lead oxide.

DETERMINATION OF ZINC OXIDE

To the filtrate from the lead sulphate in the determination of lead oxide add 8 grams of pottasium hydroxide. Bring to boil and add 4 grams of C. P. sodium potassium tratrate (Rochelle salts) and boil until solution clears. Electrolyze for zinc on copper plated platinum electrode. The current should be turned on when the solution is placed about the electrodes, otherwise the copper plating will be dissolved. Allow to electrolyze with one lamp for two hours and then step up the current for two hours more. The zinc deposits on the negative electrode as metallic zinc, but should be reported as zinc oxide.

Weight of zinc times the factor 1.245 times 2, times 100, equals per cent. of zinc oxide.

DETECTION OF CHROMATES AND PRUSSIAN BLUE

Boil a small amount of pigment with a saturated solution of sodium carbonate. Dilute and filter. Make the filtrate acid with acetic acid and divide into two portions. To one portion add a few drops of a 10 per cent. solution of lead acetate. A yellow precipitate indicated the presence of chromates. To the other portion of filtrate add a few drops of hydrochloric acid and a few drops of ferric chloride, then add a few drops of ferrous sulphate solution. A characteristic blue color is obtained if Prussian blue is present in the pigment.

SEE NOTE—PART IV.

Method of Analysis of Standard Black for Structural Steel and Iron

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF FINENESS AND SHADE

These tests are conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF CARBON

Weigh .5 gram of the pigment into a 250 c. c. beaker, add 20 c. c. nitric acid (Sp. Gr. 1.2) and four drops of hydrogen peroxide (free from acetanilid and organic compounds), preferably "Dioxygen." Bring to boil and add 25 c. c. of water and boil 5 minutes. Filter through a platinum combustion boat into a bell jar containing a 250 c. c. beaker to catch the filtrate, using suction. Wash the residue with water containing 5 c. c. nitric acid (Sp. Gr. 1.42) to 1 liter of water, and finally several times with water. Burn the carbon in a combustion furnace. The weight of carbon dioxide obtained × .2727 divided by .5×100 equals per cent. carbon in the pigment.

Note: In some cases traces of the oil will remain in the pigment. This is due to it being partially oxidized or to similar causes. In this case the combustion of the "carbon" will give high results.

DETERMINATION OF LEAD

The filtrate in the beaker in the bell jar is electrolyzed for lead with one lamp in the circuit for from four to six hours. Weight of lead peroxide × .8661 divided by .5×100 equals the per cent. of lead in the pigment.

See Note—Part IV.

Method of Analysis of Samples Taken from Painters' Buckets

When freight and refrigerator cars are being built for the Pennsylvania Railroad by car manufacturing companies and painted by them, the paste is sampled by the inspector. The samples are then sent to the Laboratory and reported as accepted before the manufacturers are allowed to thin the paste for use. This applies to the standard freight car color, standard black and refrigerator car color. The paste must then be mixed ready for use according to instructions.

In order to see that these instructions are followed, inspectors take samples of the paint mixed ready for use from the painters' buckets and send these samples to the Laboratory where the proportions of pigment, oil and volatile are determined. The operations of analysis are carried out as described under freight car color. Painters' samples are taken often enough so that the Laboratory can keep in touch with the work, four to five samples often being received in a day.

DETECTION OF PHOSPHATES

The electrolyte from the lead determination is tested for phosphates by taking one-half and boiling with ammonium molybdate solution (same solution as is used for the determination of phosphorus in steel). A yellow precipitate indicates the presence of phosphates.

DETECTION OF BARIUM AND SULPHATES

The half of the electrolyte from the lead determination not used for the detection of phosphates is tested for sulphates. Add a few drops of hydrochloric acid, take to dryness and take up with water and about 1 c. c. hydrochloric acid. Boil, add barium chloride solution. A white precipitate indicates the presence of sulphates.

The residue on the combustion boat is also tested for sulphates. Fuse the residue with sodium carbonate, add water and boil. Filter and make filtrate acid with hydrochloric acid. Bring to boil and add barium chloride solution. A white precipitate of barium sulphate indicates the presence of sulphates in the pigment. Treat the residue from fusion with hydrochloric acid and add calcium sulphate solution. A white precipitate indicates the presence of barium.

DETECTION OF CARBONATES

This test is conducted in the same manner as described in the analysis of freight car color, except that only a qualitative test need be made, as no carbonates whatever are allowed in standard black pigment.

SEE NOTE—PART IV.

Method of Analysis of Cabin Car Color

DETERMINATION OF PIGMENT, OIL AND VOLATILE

Weigh .5 gram sample into a 100 c. c. beaker and fill about half full of benzine or texene. Mix thoroughly. Filter with aid of suction through a Gooch crucible. The filter bed should be made with a rather thick layer of asbestos fibre. Wash the residue with about 20 c. c. benzine or texene and reject the filtrate. Dry crucible and contents over night on steam plate, cool and weigh. Weight of residue divided by .5 times 100 equals percentage of pigment. Weigh 2 grams paste on watch glass and place on steam plate for 4 hours. Remove from plate, cool and weigh. Loss in weight is taken as volatile. Loss in weight divided by 2 and multiplied by 100 equals percentage of volatile. The percentage of oil is obtained by subtracting from 100 the sum of the pigment and volatile.

MAUMENE'S OIL TEST

Fill a large size alundum crucible with the paste and place in position in the funnel of a bell jar, holding it in position with a rubber ring. Catch the oil in a small beaker. In order to get sufficient amount of oil, it is necessary to leave the suction on over night as the oil filters through very slowly. When 5 c. c. of oil are obtained, proceed with the test, as described under "Freight Car Color," except that the temperature rise with a boiled oil should be at least 102 degrees Fahrenheit.

FINENESS AND SHADE.

These tests are conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF PIGMENT INSOLUBLE IN CHLOROFORM

Weigh .5 gram of paste into a 250 c. c. beaker, add about 100 c. c. of chloroform, and boil for a few minutes. Filter through a Gooch crucible which has a bed prepared with asbestos. To prevent the toluidine red from precipitating out, the solution must be filtered hot and kept hot during the filtering operation. Wash out the beaker and wash the residue several times with hot chloroform. Dry crucible on hot plate, cool and weigh.

CALCULATION

The per cent. of insoluble is calculated on the per cent. of pigment found. Divide the weight of insoluble matter found by the weight of pigment found, which will give, after multiplying by 100, the per cent. insoluble in the pigment, as in each case 0.5 gram sample is used.

DETERMINATION OF ASH

Ignite the insoluble residue from the determination of insoluble in chloroform. Cool and weigh. Weight of residue in ash, which divided by weight of pigment taken (as above) after multiplying by 100 gives per cent. of ash.

DRYING TEST

Weigh 1 gram of paste and 2 grams of pure turpentine into a small evaporating dish, and grind thoroughly with test tube. Pour mixture over a small glass plate (about 3" x 6") and hold in a vertical position for exactly two minutes, then place in a horizontal position and allow to dry at 65 degrees to 80 degrees Fahrenheit temperature. This mixture should dry in 18 hours.

BLEEDING TEST

Place some of the paste on paper and draw alcoholic potash over it. The color should not turn purple. If it does, the presence of an undesirable organic color is indicated, as the desired organic color, M-nitro-P-toluidine red will show no color change upon the addition of alcoholic potash.

See Note—Part IV.

Method of Analysis of Refrigerator Car Color

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF FINENESS AND SHADE

These determinations are conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF LEAD OXIDE, ZINC OXIDE AND DETECTION OF CHROMATES

The methods described in the analysis of building paints are used.

DETERMINATION OF OPACITY

Into a small evaporating dish weigh .333 gram of Pennsylvania Railroad standard refrigerator car pigment, and into another evaporating dish weigh .333 gram of the pigment under examination. Into each dish run 1 c. c. of raw linseed oil and grind thoroughly with the end of a test tube. At one end of a microscope slide glue a square cover glass. At the edge of the cover glass place a drop of the mixed standard and the sample, side by side, then place another cover glass so that one end rests upon the cover glass glued to the slide, while the other end rests upon the slide itself, the cover glass thus covering the two drops. Now press the cover glass tightly to distribute the drops and hold slide toward the light. The relative opacity can be readily noticed and the opacity of the sample should equal that of the standard.

Note: The above test disregards the working, brushing and other practical properties of the paint. It is desirable to check this test for opacity by means of a practical test, in which the paint in question is compared with a standard paint. This test is made by brushing both paints over a surface, which has been painted white and then striped with one or more black lines. The manner in which the sample obscures the black lines, as compared with the manner in which the standard sample obscures the black lines, is a measure of the opacity of the paint.

SEE NOTE—PART IV.

Method of Analysis of Telegraph Cross Arm Color

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF FINENESS AND SHADE

These tests are conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF CARBON

Weigh .5 gram sample of pigment into a 250 c. c. beaker, add about 15 c. c. nitric acid, C. P. (Sp. Gr. 1.42) and about 25 c. c. of water. Bring to boil and then filter into a platinum combustion boat, washing well with hot water. Discard filtrate. Burn the residue in a combustion furnace, as described in the method of determining carbon in steel, the apparatus and operation being the same. Weight of carbon dioxide $\times$ .2727 divided by $.5 \times 100$ equals per cent. of carbon.

The undesirable constituents which may be present are tested for in the usual way.

SEE NOTE—PART IV.

Method of Analysis of White Lead

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF WHITE LEAD AND IMPURITIES IN PIGMENT

Weigh .2 gram pigment, well ground and mixed, into a 250 c. c. beaker. Add 20 c. c. concentrated nitric acid and about 25 c. c. water. Cover with a watch glass and stand on steam plate one-half hour when all should be dissolved. If a residue remains, filter off and analyze for barium sulphate as described in the analysis of freight car color. Electrolyze filtrate for lead which deposits on the positive electrode as lead peroxide.

Weight of lead peroxide × 1.0787 divided by $.2 \times 100$ equals per cent. white lead or basic lead carbonate. The factor 1.0787 is an empirical number obtained by a long series of experiments carried out in our laboratory with samples of the best white lead obtainable, and proved to be more satisfactory than the theoretical factor which it approximates closely.

See Note—Part IV.

Method of Analysis of Passenger Equipment Car Truck Color

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF FINENESS AND SHADE

These tests are conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF LEAD OXIDE

Weigh .5 gram of well ground pigment into a 250 c .c beaker, add 15 c. c. cencentrated nitric acid and about 40 c. c. of water. Cover with a watch glass and stand on steam plate. Remove from steam plate and boil vigorously on gauze for about one minute, filter, wash thoroughly with hot water and electrolyze filtrate for lead peroxide on the positive electrode. The residue is rejected. Any manganese present may be detected by the color of the electrolyte and must be corrected for. Weight of lead peroxide × the factor 0.93302 divided by 5×100 equals per cent. lead oxide.

DETECTION OF CHROMATES AND PRUSSIAN BLUE

These constituents are tested for in the same manner as described in the analysis of building paints.

SEE NOTE—PART IV.

Method of Analysis of Red Lead Lute

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF RED LEAD

Weigh .2 gram of the well ground sample into a 250 c. c. beaker. Add 15 c. c. of concentrated nitric acid and about 4 drops of hydrogen peroxide. Boil vigorously for several minutes, filter, wash with water containing 5 c. c. of concentrated nitric acid to 1 liter of water, and then wash with water. Electrolyze the filtrate for lead, which deposits on the positive electrode as lead peroxide. Reject the residue. Weight of lead proxide × factor .9553 divided by .2×100 equals per cent. of red lead.

See Note—Part IV.

Method of Analysis of Black Lute

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF ASH AND CARBON

Weigh .5 gram sample into a small silica crucible. Ignite strongly, cool and weigh as ash. If the per cent. of ash is too high, determine the carbon directly, carrying out the operation exactly as described in the analysis of standard black, except that .2 gram sample is used.

DETECTION OF PHOSPHORUS

Place a small amount of pigment into a beaker, add dilute nitric acid and boil. Filter. Neutralize the filtrate with ammonium hydroxide, then make slightly acid with nitric acid. Add about 25 c. c. ammonium molybdate solution, as used for the determination of phosphorus in steel, and stir for several minutes. A yellow precipitate shows the presence of phosphorus which indicates that bone black has been used in the pigment.

See Note—Part IV.

Method of Analysis of Indian Red

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color.

DETERMINATION OF SESQUIOXIDE OF IRON

The method described in the analysis of freight car color is used, except a .2 gram sample is taken instead of a .5 gram sample, and calculations made accordingly.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF FINENESS AND SHADE

These tests are conducted in the same manner as described in the analysis of freight car color.

SEE NOTE—PART IV.

Monthly Samples of Japan for Approval

METHOD FOR THE DETERMINATION OF VOLATILE MATERIAL

Weigh a five-gram sample into a weighed 100 c. c. beaker, allow to stand on the steam plate over night, cool and weigh. The loss in weight divided by the weight of the sample and multiplied by 100 is taken as the percentage of volatile material.

Note the consistency of the residue in the beaker.

METHOD FOR THE APPROXIMATE DETERMINATION OF TURPENTINE AND BENZINE IN THE VOLATILE MATERIAL, OR POLYBERIZATION TEST

Add with a pipette 10 c. c. of the sample to a four ounce distilling flask, the neck of which from the bulb to the delivery tube is 2½ inches in length, and place in position in a Liebig condenser. Distill, catching the distillate in a 50 c. c. graduated comparison tube. The heat must be applied gently to avoid bumping and splashing of the sample into the condenser. Heat until the first yellowish drops appear in the distillate, as these indicate all the "benzine" and turpentine are distilled and the oil is breaking down. Stopper and stand the tube vertically for five minutes and read the volume in tenths of a c. c. Holding the tube under cold running water, add 30 to 35 c. c. sulphuric acid, C. P., 1.84 Sp. Gr. Care must be taken to add the acid slowly, especially at first, to keep the contents of the tube cold and never to point the tube towards any one, as occasionally the heat in steam generated will "blow out" the contents of the tube. Stopper and shake the tube vigorously. On standing over night the turpentine will remain with the acid as a brownish red heavy liquid while material like "benzine" will rise as a clear insoluble layer. Read the volume of the insoluble layer in tenths of a c. c. Calculate the percentage of turpentine in the distillate, taking account of the fact that turpentine contains several per cent. of hydrocarbons, like cymene which separates with the "benzine" in this test, and for which a correction must be made. The remainder of the distillate is called "benzine" and the percentage reported.

FIFTEEN PER CENT. MIXING AND DRYING TEST

Measure 6 c. c. of the sample of japan and 34 c. c. of raw linseed oil into a 4-ounce vial. Swirl to mix, but do not shake vigorously on account of frothing. Pour some of the mixture over a glass plate (3 inches wide by 6 inches long) forming an even film. Place in a vertical position and dry at room temperature. Note the time required for the film to become dry, which should be in about 4 hours.

Allow the remainder of the mixture to stand over night, when note if separation has occurred.

HALF AND HALF DRYING TEST

Add 2 c. c. of the sample of japan and 2 c. c. of turpentine to a small evaporating dish, measuring with a pipette. Mix well by stirring with a small test tube and pour over a glass plate, forming an even film. Place in a vertical position and dry at room temperature. Note the time required for the film to become dry.

COACH DRYING TEST

Add 2 c. c. of the sample of japan to a small evaporating dish, measuring with a pipette. Add from burettes .57 c. c. of raw linseed oil and 2.66 c. c. turpentine, mix well by stirring with a small test tube, and pour over a glass plate, forming an even film. Place in a vertical position and dry at room temperature, note the time required for the film to become dry.

SHIPMENT SAMPLES OF JAPAN

The volatile material, turpentine and "benzine" are determined and the 15 per cent. mixing and drying test is made the same as described under Monthly Samples of Japan for Approval.

See Note—Part IV.

Method of Analysis of Passenger Car Color

DETERMINATION OF PIGMENT, OIL AND VOLATILE

These constituents are determined in the same manner as described in the analysis of freight car color, except to find volatile subtract the sum of pigment, oil and water from 100 per cent.

MAUMENE'S OIL TEST

This test is conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF FINENESS AND SHADE

These determinations are conducted in the same manner as described in the analysis of freight car color.

DETERMINATION OF SESQUIOXIDE OF IRON

Sesquioxide of iron is determined in the same manner as described in the analysis of freight car color, except that a 0.2 gram sample is used and the sample ignited in a procelain crucible before dissolving. Calculations are made accordingly. The purpose of igniting is to drive off the organic matter present.

DETERMINATION OF CALCIUM CARBONATE

Calcium carbonate is determined in the same manner as described in the analysis of freight car color.

DETERMINATION OF WATER

Into a 6-ounce wide-mouth Erlenmeyer flask place about 1 gram anhydrous copper sulphate and weigh. Then weigh into a flask 5 grams of the paste under examination. Fill with gasoline, allow to settle, decant the supernatant liquid, and weigh dry as described in the analysis of freight car color.

Alcohol must not be used in this determination because of the water it contains. Subtract from the weight of pigment thus obtained the weight of pigment obtained in determining the pigment and oil in the usual way. The difference is the weight of water which has been absorbed by the anhydrous copper sulphate.

Anhydrous copper sulphate is made by heating crystallized copper sulphate over a flame until the water is driven off, shown by the copper sulphate becoming white.

SEE NOTE—PART IV.

Spar Varnish

NOTE: These methods for analysis of spar varnish are based largely upon the Bureau of Aircraft Production Specification for Spar Varnish for Use on Airplanes. This specification differs from most varnish specifications in that it specifies performance instead of composition.

1. *Color.*

State color.

2. *Gas test.*

Pour varnish on glass and allow to dry in a vertical position at top of hood where gas fumes from laboratory will cause varnish to "craze" if it it not gas proof. If varnish does not "craze," duplicate test.

3. *Water test.*

Pour on glass and allow to dry in laboratory. Examine for "crazing" by gas fumes. After 48 hours put in water and note condition at end of 48 hours. (All wood oil will not turn white, linseed oil will turn white.)

4. *Kauri solution test.*

Determine total non-volatile. Weigh 2 grams by difference in a pint friction top lid. Dry 3 hours at 100° C. Cool and weigh. Loss is volatile thinners.

Make Kauri solution by melting No. 1 Kauri gum until 25% of gum is distilled off. Dissolve residue Kauri gum in 2 parts by weight of turpentine by heating the gum to about 300° F. and adding the turpentine and weighing when cool and adding the final small amounts of turpentine.

Take 100 grams of varnish and 50% Kauri solution of the weight of the non-volatile of the varnish. For example if non-volatile is 44%, take 22 grams Kauri solution. Mix, flow on 100 lb. tin sheet, drain 1 hour, bake at 100° C. for 5 hours, cool 1 hour, and bend at 70° F. over a ⅛" rod. Film should not show cracks under a shaded electric light.

A spar varnish that stands this test must be at least 33 gallons of oil to 100 lbs. of gum, and consequently will be very durable.

Shorter oil varnishes of less durability will stand a less percentage of Kauri gum solution.

Report percentage of non-volatile.

5. *Drying.*

Examine glass being prepared in "3" water test," by seeing how long drying is required before a piece of lint can be touched to the film and then blown off with the breath. Record this time as "drying dust free." See how long the film of varnish requires for drying hard. Record this as "drying hard."

6. *Additional tests that are desirable.*

(a) Practical test made by using the varnish.

(b) Viscosity. Test viscosity with Scott Viscosimeter.

(c) Ash.

Burn 20 grams in a porcelain evaporating dish and ash residue in muffle. Weigh ash. Dissolve ash in 2 c. c. HNO_3 plus 20 c. c. water plus a few drops H_2O_2. Filter and make up to 100 c. c. Divide in half and run lead in one half by making ammoniacial and then adding acetic acid until just acid, then 2 c. c. more. Clear with 1 drop of H_2O_2 and run lead by dichromate method as in analysis of white lead. Run calcium in filtrate. Make alkaline with NH_4OH, heat to boiling, precipitate with an excess ammonium oxalate, filter and wash with cold water. Wash precipitate back in beaker, add 30 c. c. of 1:2 by volume of H_2SO_4 (1.84) through paper. Dilute to 100 c. c. and heat nearly to boiling and titrate with approximately $N/_{20}$ $KMnO_4$.

1 c. c. $N/_{20}$ $KMnO_4$ is equivalent .00250 $CaCO_3$.

1 c. c. $N/_{20}$ $KMnO_4$ is equivalent .001402 CaO.

(In general 25 times the CaO is the amount of rosin taken when limed rosin is used.)

Manganese.

Take the other half of the filtrate, add 15 c. c. HNO_3, boil, add sodium bismuthate to a color, clear with a few drops of saturated KNO_2, boil to expel fumes of NO_2, cool and add about .5 gram sodium bismuthate, and filter through Gooch. Titrate filtrate with sodium arsenite as in manganese in steel.

(d) Acid number.

28 grams varnish dissolved in 100 c. c. of a neutral mixture of equal parts of benzine and alcohol. Add phenolphthalein and titrate with $N/_2$ alkali.

$\frac{cc \times 28}{\text{sample}}$ is equivalent to acid number.

$\frac{\text{acid number}}{2}$ is equal to oleic acid.

Methods for Rubber Analysis

GENERAL

Make the analysis upon the insulation after vulcanization and before the saturation of the braid. Wipe the insulation thoroughly with a damp cloth to remove any adhering material, but do not remove waxy hydrocarbons from the surface.

Perform all determinations in duplicate and take the average value arbitrarily as the true value. Duplicate determinations must check within .10 to 1%.

Make blanks on all determinations and deduct accordingly.

SAMPLE

Remove the insulation entirely from sufficient wire to give a sample weighing about 25 grams. Cut this into small strips and grind slowly in a No. 0 Enterprise coffee mill. Adjust the grinder so that it all, on regrinding, will pass through a 20 mesh sieve and at least 60 per cent. will be retained on a 40 mesh sieve. The wires of the sieve shall be evenly spaced in both directions and shall be of 0.016 and 0.010 inch diameter in the 20 and 40 mesh sieves respectively. Remove with a strong magnet any metal that may have come from the grinder and thoroughly mix the sample.

EXTRACTION APPARATUS

The extraction apparatus used for the acetone and chloroform extractions shall consist of a siphon cup 28 m. m. inside diameter, 70 m. m. long, in which is placed a 22 m. m. acetone extracted paper thimble, to hold the sample. This is suspended in a flask or tube with a reflux condenser. It shall be heated so that the period of filling an empty siphon cup with acetone and completely emptying it will be between 2½ and 3½ minutes.

PREPARATION OF REAGENTS

Acetone shall be freshly distilled over anhydrous K_2CO_3, using the fraction 56-57 C.

Alcoholic KOH solution shall be of normal strength and shall be freshly made by dissolving the proper amount of KOH (purified by alcohol) in 95 per cent, alcohol, which has been distilled over KOH. The solution shall be allowed to stand for 24 hours and only the clear liquid used.

Chloroform shall be pure and freshly distilled.
Carbon tetrachlorides shall be pure and freshly distilled.
Regents not otherwise specified shall be C. P.

ACETONE EXTRACTION

1. Extract with acetone for eight hours, a 2 gm. sample. If the extraction cannot be continuous, keep the sample in either an atmosphere of acetone or nitrogen. The extraction flask must have been weighed or transfer the extract to a weighed flask, using hot chloroform to rinse the extraction flask or tube. Distill off the reagents and dry the flask and contents for four hours at about 95° C. Desiccate until cool and weigh. Continue to dry for two-hour periods until constant weight is obtained. (See Par. 2.) (Residue See Par. 6.)

UNSAPONIFIABLE MATERIAL

2. Add to the acetone extract (see paragraph 1) 50 c. c. alcoholic KOH solution, boil under a reflux condenser for two hours and evaporate to dryness, removing all alcohol. Add a few c. c. ether and a few c. c. water, heat, transfer to a separatory funnel, wash out the flask with warm water, and when cool, finally with 25 c. c. of ether in two portions. The water volume should be 100 c. c. Shake with a rotary motion for two minutes and allow the solution to separate thoroughly. Draw off the aqueous solution into a second funnel, leaving in the first funnel the ethereal solution and any flocculent material that may be present. Again rinse the flask with 20 c. c. of ether and add it to the aqueous solution; shake for two minutes and when separated, draw off the aqueous solution and unite in the first funnel the ethereal solutions and any flocculent material. Repeat, shaking with 20 c. c. portions of ether until the extraction is complete, shaking at least four times. Wash the ethereal solution with water until it has been washed twice after it shows no alkaline reaction, retaining with the ethereal solution any flocculent material. (See Par. 5.) Filter the ethereal solution from the flocculent material through a small pellet of extracted cotton, into a weighed flask, washing first with ether and subsequently with hot chloroform, using this to rinse the separatory funnel. Evaporate the solvents and dry the extracts to constant weight at about 95° C., disiccate until cool and weigh. (See Par. 3.)

HYDROCARBONS (A)

3. Add 50 c. c. absolute alcohol to the unsaponifiable material (P2) and warm until solution is as complete as possible. Cool the solution to -4 or -5° C., and maintain at this temperature for one

hour by packing the flask in a mixture of ice and salt. Filter out the waxy hydrocarbons, using a funnel packed with ice and salt, and apply suction if necessary. Wash the flask and filter with about 25 c. c. of 95 per cent. alcohol which has been previously cooled to the same temperature. Catch the filtrate in a flask which is afterwards cooled to -4 or -5° C. to make sure that all possible waxy hydrocarbons have been removed and refilter if necessary. (See Par. 6.) Dissolve the residue on the filter paper with hot chloroform into the original flask. Evaporate the chloroform and dry the flask to constant weight at about 95° C.; cool in a desiccator and weigh.

HYDROCARBONS (B)

4. Evaporate the alcohol from the flask containing the alcohol-soluble unsaponifiable material, (3) add 25 c. c. carbon tetrachloride and transfer to a separatory funnel. Shake with concentrated H_2SO_4, drain off the discolored acid and repeat with fresh portions of acid until there is no longer any discoloration. After drawing off all the acid, wash the carbon tetrachloride solution with repeated portions of water until all traces of acid are removed. Transfer the carbon tetrachloride to a weighed flask; evaporate off the solvent and dry the flask to constant weight at about 95° C. Cool in a desiccator and weigh.

FREE SULPHUR

5. Evaporate the ether from the aqueous solution, first washing from the ethereal separation of the unsaponifiable material. (See Par. 2.) Add 25 c. c. bromine water, heat for one hour, making slightly acid with HCl, heat until the excess of bromine is driven off, filter and wash, making a volume of 200 c. c. Heat to boiling and add slowly a slight excess of hot 10 per cent. $BaCl_2$ solution. Allow to stand over night, filter, wash, ignite, weigh the $BaSO_4$ and calculate to sulphur.

CHLOROFORM EXTRACTION

6. Extract the residue from the acetone extraction, (See Par. 1) without removing the acetone on it for four hours with chloroform, either using a weighed flask or transfer the extract to one. Distill off the solvent and dry the flask, contents, for two hours at about 95° C. Desiccate until cool and weigh. Continue to dry for one-hour periods until constant weight is obtained. (Residue See Par. 7.)

ALCOHOLIC POTASH SOLUTION.

7. Dry the residue from the chloroform extraction (P6) at 50-60° C. Put into a 200 c. c. Erlenmeyer flask with 50 c. c. alco-

holic KOH solution and boil for four hours under a reflux condenser. Filter the solution into a beaker and wash twice, using each time 25 c. c. hot alcohol, and then wash thoroughly with hot water. Evaporate the solution to approximate dryness, take up in warm water and transfer to a separatory funnel. Acidify with 15 c. c. of 5N HCl, using this to rinse the beaker. Add sufficient water to make the bulk of the solution 100 c. c. When cool add 40 c. c. ether, using it to rinse the beaker in 20 c. c. portions. Shake the aqueous and ethereal solutions thoroughly. After complete separation, draw off the aqueous solution and treat in another separatory funnel, with a fresh 20 c. c. portion of ether. Continue to shake the aqueous solution with fresh portion of ether until a colorless portion has been optained, then shake out twice more. Unite the ethereal solutions and wash with successive portions of water, continuing twice after the water shows no acid reaction. Filter through a plug of extracted cotton into a tared flask, wash the filter and funnel with ether, evaporate the ether without boiling and dry the residue to constant weight at about 95° C. Cool in a desiccator and weigh.

ASH

8. Wrap a 1-gram sample in an 11 c. m. ashless filter paper. Extract with acetone for four hours and place, still wrapped in the filter paper, in a weighted porcelain crucible to which is fitted a wide asbestos collar. Heat at the lowest possible temperature over a Bunsen burner, distilling the organic matter without ignition. Finally volatilize and condensed material over a low flame. Desiccate until cool and weigh. (See Par. 9.)

SULPHUR IN ASH

9. This may be done by either method. Add a few drops of concentrated HNO_3 saturated with bromine, to the ash, (See Par. 8) stir with a glass rod and evaporate off the excess of acid. Add 5 g. fusion mixture, equal parts, Na_2CO_3 and KNO_3, and fuse. Cool, dissolve and melt in water, filter (See Par. 1) and acidify slightly the filtrate with HCl. Heat to boiling and add slowly a slight excess of hot 10 per cent. $BaCl_2$ solution, allow to stand over night, filter, wash, ignite, weigh the $BaSO_4$ and calculate to sulphur.

10. Add to the ignition dish and ash (See Par. 8) placed in a casserole 25 c. c. of bromine water, cover with water and bring to a boil. Acidify slightly with HCl, heat to drive off the bromine and filter. Ignite the insoluble residue in a platinum crucible and fuse with 3 g. fusion mixture, equal parts Na_2CO_3 and KNO_3. Dissolve the melt in the water, filter, (See Par. 1) acidify the filtrate slightly

with HCl and add to the preceding filtrate. Heat to boiling and add slowly a slight excess of hot 10 per cent. $BaCl_2$ solution. Allow to stand over night, filter, wash, ignite, weigh the $BaSO_4$ and calculate to sulphur.

SULPHUR AS BARYTES

11. Wash back into the original beaker with hot water the insoluble residue left after filtering the melt. (See Pars. 9 or 10.) Acidify with HCl and heat until as much as possible is dissolved. Filter through the same filter paper and nearly neutralize with Na_2CO_3, leaving the solution slightly acid. Saturate the solution with H_2S when the lead sulphide has settled, filter and wash, making the volume to about 200 c. c. Heat to boiling and add slowly a slight excess of 10 per cent. H_2SO_4. Allow to stand over night, filter, wash, ignite, weigh the $BaSO_4$ and calculate to sulphur.

TOTAL SULPHUR

12. This may be done by either method. Mix a .5 gm. sample with 4 gm. Na_2O and 6 gm. K_2CO_3 in a dry 15 c. c. iron crucible. Cover and heat gradually until the mixture fuses, proceeding cautiously as rapid heating will cause an explosion, and then bring to quiet fusion, for 15 to 20 minutes. Apply the heat so as to avoid the contamination with sulphur fumes. Rotate the crucible while the melt solidifies. When cool, put crucible and cover into a casserole containing 200 c. c. of water; add 5-10 c. c. bromine water and boil until the melt is dissolved. Allow the precipitate to settle, decant the liquid through a thick filter and wash the residue with hot water, acidify the filtrate with HCl evaporate to dryness, add 2 c. c. concentrated HCl, take up in water, filter and wash, making the total volume about 400 c. c. Heat to boiling and add slowly a slight excess of hot 10 per cent, $BaCl_2$ solution. Allow to stand over night, filter, wash, ignite, weigh the $BaSO_4$ and calculate to sulphur.

CARBON

13. Heat about 1 gm. with 30 c. c. concentrated HNO_3 and 15 c. c. water. A black insoluble residue indicates the presence of carbon.

SPECIFIC GRAVITY

14. The specific gravity shall be the ratio of the weight of a given volume of the rubber, to the weight of an equal volume of water, both at 20° C. Cut strips of the largest applicable size from the conductor and use about 5 gm. for the sample. Determine the specific gravity in the usual manner by the means of the specific gravity bottle. Care must be taken that no air bubbles adhere to

the sample, removing these by subjecting the sample in the bottle to a vacuum if necessary.

STATEMENT OF RESULTS

Calculate the results to percentages. Report the acetone extract as such. Add the percentages of Hydrocarbons A and B and report the results as Waxy Hydrocarbons. Subtract the sum of the percentages of Waxy Hydrocarbons and free sulphur from the acetone extract, and report the result as Rubber Extract. The difference between the unsaponifiable material and the Waxy Hydrocarbons is reported as the Unsaponifiable Acetone Extract. The difference between the total Acetone Extract, and the sum of the free sulphur and the unsaponifiable material is reported as the Saponifiable Acetone Extract. Subtract the difference between the percentages of the sulphur in ash, from the ash and report the result as Ash Corrected, or Free Sulphur. Subtract the sum of the percentages of waxy hydrocarbons, ash corrected and total sulphur, from 100 and report the result as Rubber, by difference. Note: In case the chloroform and alcoholic potash extractions do not come within the limits of the specification, this form of calculation may be incorrect. Report also the following:

Color of Acetone Extract, (60 c. c. Vol.).
Fluorescence in Acetone Extract Solution (present or absent).
Hydrocarbon A (consistency).
Color of Chloroform Extract, (60 c. c. Vol.).
Carbon (present or absent).

Additional Rubber Methods

FREE SULPHUR

When this is the only constituent of the acetone extract that is needed, it may be determined as follows: Add to the acetone extract 25 c. c. concentrated HNO_3 saturated with bromine and heat on the steam plate until the bromine fumes are nearly driven off. Dilute, and when cool, filter, add NaCl to the filtrate so as to have enough base to combine with the sulphur molecule. (25 c. c. of a solution 20 g. to the liter is usually an excess.) Evaporate to dryness and take up in water, make slightly acid with HCl and add $BaCl_2$, etc., as usual and determine sulphur. The difference between the free sulphur and the acetone extract is the rubber extract in material containing no waxy hydrocarbons.

TOTAL SULPHUR

Mix thoroughly 1 gm. sample with .5 gm. tartaric acid and 4-6 gm. Na_2O_2 in the combustion cartridge of the Parr Calorimeter. Fire by means of a small iron fuse wire and an electric current, dissolve the fused mass with hot water, add a few c. c. of Bromine water and after standing on the steam plate about 1 hour, filter, make acid with HCl, add $BaCl_2$, etc., as usual and determine sulphur.

ANALYSIS OF RUBBER

By A. S. & W. Co.'s Procedure.

Procedure up to and through the alcoholic potash extract according to the J. R. I. C. Report.

Wash all the residue from the alcoholic potash extract into a 300 c. c. Erlenmeyer flask and add sufficient water to make a volume of 100-125 c. c. Add 25 c. c. of HCl (1.2) and heat at 95-100° C. until all fillers soluble in HCl are removed. Decant the HCl solution through a hardened filter paper (S&S number 575, 15 c. m.) or through an Alundum crucible, using suction. Treat the residue several times with hot water, decanting the wash liquors through the filter. Boil the residue for several minutes with water and decant the solution, washing the residue with hot water. Continue boiling and washing with water until free of soluble chloride. Transfer the residue to the filter and wash with 25 c. c. alcohol. Dry for about 15 minutes at 95-100° C. and remove to a tared weighing bottle. Heat the residue ½ hour at 95-100° C., and then subject it to a suction in a vacuum desiccator for about 20 minutes and weigh. Continue heating and evacuating for shorter periods until constant weight is obtained. Determine ash on about ½ of this residue and sulphur on the remaining portion. Also determine sulphur on the ash. Sulphur determinations to be performed by the J. R. I. C. total sulphur method. Blank determinations to be made and allowed for.

Rubber Hydrocarbons:

$$\text{Per cent.} = 100 \times \frac{\text{Residue from KOH and HCI ext's.}}{\text{Wt. of Original Sample}} \left(1 - \frac{\text{Sulphur}}{\text{Residue from KOH \& HCI ext's.}} \quad \frac{\text{Ash}-\text{Sulphur}}{\text{Sample for Ash}} \right)$$

Rubber Hydrocarbons plus saponifiable, unsaponifiable acetone extract chloroform and alcholic KOH extracts equal rubber.

This calculation of rubber is in error if the material contains fillers soluble in chloroform, and in acetone which are not taken into account as waxy hydrocarbons. If the material contains substitute this is shown in the alcoholic KOH extract and this must then not be added to obtain the percentage of rubber.

It is probable that the alcoholic KOH extraction would bo better omitted from this determination when no substitutes are present.

REVISED PROCEDURE FOR FILLERS.

Wrap a 1 g. sample in filter paper (S&S 590) and extract with acetone for four hours. Transfer the residue to a tall lipped 200 c. c. beaker. Add 75 c. c. Xylol and heat at 100-110° C. until there is a clear solution, stirring occasionally. (This may require a few days.) Decant the supernatant liquid into a beaker. Add to the undisturbed residue 30 c. c. Xylol and stir. Heat for several hours, decant, unite the decanted solution and repeat this treatment until rubber is dissolved. The Xylol and benzol filtrates should be kept separate from the combined acetone and water filtrate. Transfer the residue to the filter paper with hot water. Dry at 95-100° C. and wigh. Replace filter paper in funnel, treat residue with cold (2.3) nitric acid, using 25 c. c. portions, until completely dissolved.

Note: It is essential thet the reaction at this point be complete and therefore the treatment must be repeated a number of times.

Wash with cold water until free of nitric acid and then wash with hot acetone until nothing more is dissolved, catching the acetone in a weighed beaker. Evaporate the acetone, dry at 95-100° C. and weigh. Add fillers on filter paper to ash and subtract acetone soluble matter. Subtract one per cent. as a tentative correction for proteid and other imsoluble organic matter. Determine sulphur in fillers on a separate sample and report it separately. Subtract the sulphur from the corrected result if this method is used for fillers, with a total sulphur determination to calculate rubber.

Rapid Determination of Carbon in Steel

The method used in a modification of the method published by William R. Fleming in the Iron Age, January 1st, 1914. Such modifications were made to suit the character and volume of work, where several hundred carbon determinations are made daily.

The Carbon Train is shown in Eimer & Amend's Bulletin, and consists of a high pressure oxygen tank which when received has a pressure of about 1,800 pounds per square inch. The oxygen is of sufficient purity to require no pre-heating. The pressure is reduced through a Davis-Bournonville Regulating and Reducing Valve, (see E. & A.'s Bulletin), to the pressure required for determination, which is about two pounds.

The oxygen now passes through a long glass tube of the carbon filtering type. It is filled for three inches from either end with rather coarse Calcium Chloride. The middle portion is filled with 60 mesh Soda Lime, separated from the Calcium Chloride by asbestos plugs. These two reagents remove any moisture or carbon dioxide which may have been in the oxygen.

The purified gas now passes into a fused quartz tube 30 inches long and ⅞ inch inside diameter, in which is a three-inch plug of asbestos, about one-third the distance from the out-going end. This tube is placed in a Carbon Combustion Type Electric Furnace, capable of being maintained at a temperature of 1,000 degrees Centigrade, and which can be used either on 110 volt or 220 volt circuit. The current is controlled by a Rheostat and a double-pole knife-switch protected by suitable fuses.

The oxygen with the products of combustion, now pass into a Dudley bubble-tube which is filled about one-third with 20-mesh granulated zinc, which absorbs any sulphur fumes which may arise from the compustion.

The gases now pass into a Fleming Tower which contains Phosphoric Anhydride, which removes any moisture, and then into a Fleming bulb. (E. & A.'s Bulletin.) This bulb contains layers of 20, 40 and 60-mesh Soda Lime in the bottom bulb, and Phosphoric Anhydride in the upper bulb. The openings of the Fleming Bulbs and Towers are protected by plugs of asbestos to prevent any Phosphoric Anhydride or Soda Lime being carried over mechanically. The carbon dioxide from the combustion is retained by the Soda Lime.

METHOD OF DETERMINATION.

The furnace being heated and maintained at a temperature of 1000° C., a current of oxygen equal to a flow of about 350 c. c. per minute is passed through the train for a sufficient time to drive out all air, and a blank determination is run at the same rate. In weighing the Fleming Bulb it is best to use another bulb similarly charged as a counterpoise. The valves in the absorption bulb are always closed during weighing, and opened when connected with the train again.

Weigh 1.5 grams of drillings of about uniform size and spread out in an Alundum Boat filled with R. R. Alundum, free from moisture and carbonates, cover sample completely with Alundum and place in quartz tube about one-third of the distance from the optgoing end, or in the hottest part of the tube. Stopper tube, and turn on oxygen to a flow of about 350 c. c. per minute. The drillings will be burned in about 1.5 to 2 minutes. Continue the flow of oxygen for about four minutes longer to wash out all carbon dioxide, disconnect bulb, close valves and weigh. The increase in weight multiplied by 2 and divided by 11 gives the percentage of carbon in the steel.

Some steels and some coarse drillings of the same steel require longer time for the combustion.

APPARATUS AND REAGENTS.

1 Regulating and Reducing Valve.
1 Carbon filtering tube, 11 x ⅝ inch.
1 Mercury pressure gauge.
1 Silica Tube 30 in. long, ⅞ in. inside diameter.
2 Rubber stoppers to fit above tube.
1 Phosphoric Anhydride jar.
1 Fleming absorption tube.
1 Dudley bubble-tube 3½ in.
1 Dudley bubble-tube 2½ in.
1 Calcium Chloride drying tube, straight.
Alundum Boats, 3½ x ½ in., No. 3729.
1 Large Nickel Boat, to ignite Alundum in Muffle.
R. R. Alundum.
Soda-Lime, 20, 40 and 60-mesh.
Phosphoric Anhydride.
Rubber Tubing.
1 Carbou Combustion Furnace.
1 Reostat, for above furnace.

Colorimetric Method of Determining Carbon in Fire-Box Steel. Eggertz Method

APPARATUS

Test Tubes: 6 by ¾ inches; wash bottle with rubber bulb attachment such as is used with an atomizer.

Burettes: Comparison tubes of about 20 to 30 c. c. capacity, 12 by ½ inches, graduated to .10 or .15 c. c.

Water bath for heating test tubes, a copper pot 7 inches in diameter, 6 inches tall, fitted with a circular rack of sheet copper holding 18 test tubes.

Box or camera for making comparisons. The one used at this Laboratory is made of ½ inch material, 8 by 4½ inches at the larger end, 6 by 4½ inches at the smaller end, and 24 inches long, outside measurements. It is painted black, mounted on a stand, a piece of ground glass is inserted within the camera 1¼ inches from the smaller end. A row of ⅝ inch holes ¼ inch apart are bored through the top of the camera about two inches from the smaller end.

OPERATION.

Weigh .2 gram sample in the test tubes and also sample of standard steels representing both the lower and upper limits of carbon allowed by the specifications. These standard steels must be the same kind of steel as the samples. Place rack with the test tubes in cold water of such depth that the bottom of the test tubes are at least 1½ inches under water. This to prevent too violent action on the addition of the acid. A copper pot similar to the hot water bath may be used. Add 3 c. c. nitric acid, 1.20 specific gravity, free from hydrochloric acid and chlorine, to each test tube and allow to stand until action ceases. Cover the test tubes with small watch glasses or glass bulbs and transfer to the hot water bath, the water in which has been previously brought to boiling. Covering the test tubes prevents the formation of iron oxide on the sides of the tubes, which, if present, would interfere with the color comparison. Boil for 20 minutes (error will be introduced by boiling too long) and transfer to the cold water bath. Keep the solutions in the dark, when cold, transfer first the solutions of the standard steel to comparison tubes, washing out the test tubes with water, which add to the solutions and dilute to 6 c. c. Mix well and place these tubes in the holes of the camera, so that there will be one hole between those occupied by the tubes with the standard solutions. Treat the sample solutions in the same manner as the standards, diluting the comparison tubes to 6 c. c. and placing each in turn between the tubes containing the standards in the camera. If the color of the sample solution is not lighter than that of the lower standard nor darker than that of the upper standard the sample is considered satisfactory.

A good way to handle this method is to work with four comparison tubes, two for the standards and two for the steel samples. After pouring from the test tube to the comparison tube rinse out the test tube once or twice with water from a burette, as it can be done more accurately in this way. Place the thumb, protected by a rubber finger cot, over the mouth of the tubo and mix by inverting the tube several times. The presence of air bubbles in the solution will prevent a comparison being made at once. To save time another sample should be prepared in the fourth tube after which the first sample solution will be clear. Observe the same procedure with the remaining samples.

Copper in Steel

Weigh 5 grams into a 600 to 700 c. c. beaker and add 150 to 200 c. c. water, plus 12 to 15 c. c. of concentrated H_2SO_4. Allow the action to proceed in the cold until most of the steel is in solution, or heat carefully over a low flame. Finally boil to a complete solution. Add about 3 grams of sodium thiosulphate and continue to boil until the milky solution thus produced clears up, or, at the most, is only slightly opalescent. This will require from 12 to 15 minutes vigorous boiling. Filter on a rapid filter paper, washing with hot water. Remove filter paper and wash precipitate into a 250 c. c. beaker by means of a stream of water directed from the wash bottle into the paper. Add about 5 to 10 c. c. of agua regia and about 5 c. c. concentrated H_2SO_4. Evaporate to fumes. Add water and a slight excess of ammonia. Allow the iron precipitate to collect, filter and wash with 2 per cent. ammonia water and add 10 to 12 c. c. concentrated H_2SO_4. Dilute to 400-500 c. c. water, boil and again precipitate with sodium thiosulphate. Filter, wash, ignite while moist and weigh as copper oxide (CuO).

Factor = 79.87 per cent. Cu.

NOTE: The contents of the crucible may be treated with about 2 to 3 c. c. of strong nitric acid, and warm to complete solution on the steam plate, make ammoniacal, filter into a Nessler tube and compare with the standard of known strength. If the results are high, the contents of the tube is returned to the beaker, made acid with H_2SO_4, and the copper determined by electrolysis.

Chromium and Vanadium in Steel

CHROMIUM

1 gram sample—50 c. c. H_2O plus 12 c. c. H_2SO_4 (1.84) plus 15 c. c. HNO_3 (1.42). Boil to remove oxides of N, then add 50 c. c. H_2O and sufficient $KMnO_4$ solution (phosphorus oxidizing solution)—about 10 c. c.—to give an excess of MnO_2 after boiling 20 minutes. Cool, filter through asbestos, and wash 3 times with cold water. Dilute filtrate to 300 c. c. add 30 c. c. of dilute H_2SO (1 of acid to 3 of water by volume) and titrate with an excess of ferrous sulphate solution until all brown tints are gone. (Ferrous sulphate approximately N/$_{20}$ 1 c. c. equals .000866 Cr.). Titrate excess ferrous sulphate with approximately N/$_{20}$ $KMnO_4$ (1.58 grams per 1000 c. c.).

$$\frac{\text{Ferrous sulphate} \times \text{factor} \times 100}{\text{sample}} \text{ is equivalent to \% Cr.}$$

Iron value × .3103 is equivalent to Cr. value.

VANADIUM

Take solution from Chromium determination and add 2 drops ferrous sulphate solution to destroy permanganate color, then 1 c. c. 1/10 % $K_3FeC_6N_6$ and titrate slowly with ferrous sulphate to a green color.

Iron value × .914 is equivalent to Va value.

1 c. c. N/$_{20}$ ferrous sulphate is equivalent to .00255 Va.

Determination of Manganese in Steel

AMMONIUM PERSULPHATE METHOD.

PROCESS.

Weigh 0.1 gram sample into a 250 c. c. Erlenmeyer flask, add 15 c. c. dissolving solution and boil to complete solution and expulsion of nitrous fumes. Dilute to about 75 c. c. with hot distilled water, bring to boil, remove from heat and add 15 c. c. ammonium persulphate solution. Allow to stand about a minute to obtain permanganate color. Cool in water bath and titrate while cold with standard solution of sodium arsenite.

SOLUTIONS.

Sodium Arsenite. Weigh 0.6733 gram of As_2O_3 and 2 grams Na_2CO_3 into a 250 c. c. beaker, add about 100 c. c. water and boil until all is dissolved. Boil until CO_2 is expelled. Dilute to 2 liters with water. Standardize against a standard steel. Or use 0.84 grams sodium arsenite to 2 liters H_2O. 1 c. c. should equal .0001 gram Mn.

DISSOLVING SOLUTION.

1500 c. c. Water.
500 c. c. Nitric Acid (Sp. Gr. 1.42).
70 c. c. Sulphuric Acid (Sp. Gr. 1.84).
2.76 grams Silver Nitrate.

AMMONIUM PERSULPHATE SOLUTION.

Dissolve 100 grams ammonium persulphate in 2 liters water.

NOTES AND PRECAUTIONS.

When working with steels which are difficultly soluble in the dissolving solution, such as chromo-nickel and chromo-vanadium steels, the sample should be moistened with a few c. c. of the persulphate solution before adding the dissolving solution. This hastens the solution.

Method of Determining Manganese in Steel Containing Chromium

APPARATUS AND REAGENTS.

The apparatus required by this method needs no special comment, except, perhaps, the asbestos filter. The ordinary well-known funnel and platinum perforated cone may be used, but better the glass tube filter. Instead of using those made with constrictions, such as are usually shown in apparatus catalogues for filtering acid solutions, a tube drawn out at one end to go into the hole in the cork, and with a flat coil of platinum wire to hold the asbestos, is preferable. Those made from 1⅛ in. tubing, about 8 in. long when finished, and with the drawn-down portion about 3 in. long, work nicely. The flat platinum coil is about ⅞ in. in diameter, and has a tail of the same wire hanging down from the center of the coil, about 4 in. long to assist in removing the filter from the tube after the washing is finished.

The C. P. nitric acid is obtained in the market, and the dilute is made from it by adding distilled water.

The chlorate of potash is the commercial salt obtained in the market.

The asbestos which works best is the mineral known as actinolite. Much of the asbestos of the market packs badly under suction and retards filtration.

The ferrous sulphate solution is made by adding 2 liters of concentrated C. P. sulphuric acid, specific gravity 1.84, to 14 liters of distilled water in which has been dissolved 80 grams of C. P. ferrous sulphate. It is not necessary to filter. After all the reagents are together the solution is thoroughly mixed, and its strength obtained in terms of the permanganate solution. Fifty c. c. of the ferrous sulphate solution require about 15 c. c. of permanganate. Not less than three tests should be made, and they should agree within a tenth of a c. c. If not the temperatures, the burettes, the pipette, or the mixing of the solution, one or all, are at fault.

The permanganate of potash solution for titration is made as follows: To 1 liter of water add 2 grams of crystallized permanganate of potash, and allow to stand in the dark not less than a week before using. Determine the value of this solution in terms of metallic iron. For this purpose 150 to 200 mg. of iron wire or mild steel are dissolved in dilute sulphuric acid (10 c. c. of strong C. P. acid to 40 c. c. of water) in a long-necked flask. After solution is complete, boil 5 to 10 minutes, then dilute to 150 c. c., pass the liquid through the reductor, and wash, making the volume up to 200 c. c. Now titrate with the permanganate solution. It is, of course, essential that the amount of iron in the wire or soft steel should be known. The standard in use in this laboratory is a mild steel, in which the iron is known by determining carbon, phosphorus, silicon, sulphur, manganese and copper, and deducting the sum of these from 100 per cent. Not less than two determinations are made, and three are better. The figures showing the value of the permanganate solution in terms of metallic iron should agree to a hundredth of a milligram in the different determinations. A very satisfactory method of making and keeping permanganate of potash solution is as follows: Have a large glass bottle holding say eight liters, and two half the size. Paint the outside of these bottles with several coats of black paint or varnish. Fill the large bottle with the standard solution, and after it has stood a proper time, fill one of the smaller bottles from it without shaking and standardize. At the same time fill the second small bottle and refill the large one. When the first small bottle is exhausted, standardize the second one and fill the first from tho stock. When this is exhausted, standardize the first again and fill the second from stock, refilling again the stock bottle, and so on. By this means a constant supply of sufficiently matured permanganate is always available. Of course, if the consumption is very large, larger hottles or more of them may be required. Since changes of temperature affects the volume of all solutions, it is desirable that the permanganate solution should be used at the same temperature at which it was standardized. With the strength of solutions above recommended, if the permanganate is used at a temperature of 20 degrees F. different from that at which it was standardized, the error amounts to less than 0.003 per cent. on a steel containing 0.50 per cent. of manganese.

OPERATION.

Put 3 grams of the fine borings in a 16-oz. Erlenmeyer flask, and add 50 c. c. of dilute nitric acid (Sp. Gr. 1.13). Put on the steam table or over the lamp, and as soon as solution is complete, add 100 c. c. of concentrated C. P. nitric acid (Sp. Gr. 1.42). Heat to boiling, withdraw from the fire momentarily, then add 5 grams of crushed chlorate of potash, free from manganese. Replace over the heat and boil 10 minutes; then repeat the operation with 5 grams more of chlorate. Allow to cool, then filter through an asbestos filter, and wash both flask and filter with distilled water, until the washings no longer react acid. Put the asbestos with the manganese on it back into the flask, and add 50 to 100 c. c. of ferrous sulphate solution, agitate until the manganese is all dissolved, then titrate the excess of ferrous sulphate with permanganate of potash.

CALCULATIONS.

Atomic weights used: Iron, 56; manganese, 55. Molecular formulae used: Ferrous sulphate, ($FeSO_4$); manganese dioxide, (MnO_2); sulphuric acid, (H_2SO_4); permanganate of potash, ($KMnO_4$); water, (H_2O). Reaction in ferrous sulphate solution, MnO_2 plus 2 $FeSO_4$ plus 2 H_2SO_4 equals $MnSO_4$ plus $Fe_2\ (SO_4)_3$ plus 2 H_2O.

The soft steel employed in standardizing permanganate of potash solution in this Laboratory contains 99.27 per cent. metallic iron; 0.1498 gram of this contains, therefore, (0.1498 × 0.9927) .1497 gram of metallic iron. This requires, say 42.9 c. c. permanganate solution of 1 c. c. of permanganate solution is equal to (.1487 ÷ 42.9) .003466 metallic iron. Assuming now that the manganese oxide obtained in MnO_2, it is obvious that the two molecules of $FeSO_4$ are required for reaction with one molecule of MnO_2, or the ratio between Fe and Mn in the reaction in the ferrous sulphate solution is as 112 to 55. But each c. c. of permanganate is equal to .003466 or metallic iron, and consequently (112:55: :.003466: .0017) each c. c. of permanganate is equal to .0017 of metallic manganese. Suppose now that 50 c. c. of the ferrous sulphate solution is equal to 14.9 c. c. of the permanganate solution, and that after the manganese oxide on the asbestos filter has been added to 50 c. c. of the ferrous sulphate solution, and the reaction between these is complete. It requires 4.1 c. c. of the permanganese solution to react with the excess of ferrous sulphate. It is evident that the amount of manganese in 3 grams of steel is equivalent to (14.9 — 4.1) 10.8 c. c. of the standard permanganese solution, that is to (10.8 × .0017 = .01835 gram of metallic manganese. Then if 3 grams or parts of steel contain .01835 gram of manganese, 1 gram or part would contain (.01835 ÷ 3) .00612 gram, and 100 grams or parts would contain (.00612 × 100 .612 gram or 0.612 per cent. This explanation may be briefly summarized as follows: Obtain the strength of the permanganate solution in terms of metallic iron. Then obtain the strength of the same solution in terms of metallic manganese in accordance with the reaction given above. Next find the equivalent of the amount of ferrous sulphate solution to be used in an analysis in terms of the permanganate solution. Then, after the number c. c. of permanganate solution equivalent to the manganese in the steel are obtained, multiply these by the manganese equivalent of the permanganate solution. Move the decimal point two places to the right, and divide by 3. The quotient will be the per cent. of manganese in the steel.

NOTES AND PRECAUTIONS.

This method, it will be observed, separates the manganese from the iron in the nitric acid solution by means of chlorate of potash, catches the precipitate oxide of manganese on an asbestos filter, and determines the amount of the manganese by means of the reaction between ferrous sulphate and this oxide in sulphuric acid solution, permanganate of potash being used, to measure the change in the ferrous sulphate solution, produced by the manganese oxide.

The use of dilute nitric acid to effect rapid solution of the steel, and the subsequent addition of the specified amount of concentrated acid, is found to give exactly the same results, as are obtained when the steel is dissolved in concentrated acid from the start, or, are as given, when a larger quantity of dilute acid is used to start with, followed by subsequent concentration. The saving in time by the method recommended amounts to from one to two hours.

It sometimes happens that no precipitation of manganese oxide will follow the first addition of chlorate, and even sometimes the second addition is very slow in producing a precipitate. It is hardly safe to pronounce no manganese present until quite continued boiling, and further additions of chlorate, and even more nitric acid and further boiling leave the solution quite clear. After the precipitation is once started it apparently goes on to completion without difficulty. It is not advisable to add the chlorate of potash to the boiling solution over the fire. Under certain conditions disastrous explosions follow such practice.

It occasionally happens that permanganate acid is formed in the nitric acid solution. When this is the case, the filtrate will in some cases at least be clearly pink in color, and on standing, oxide of manganese will separate. It is believed that this possible difficulty is obviated, by allowing the nitric acid solution to cool before filtration. This practice is regarded as better than adding fresh unboiled concentrated nitric acid, as has been recommended, since fresh nitric acid may contain nitrous acid, in the presence of which the separated manganese is somewhat soluble.

The length of time that the nitric acid solution with the separated manganese oxide in it, may stand without injury, seems to be considerable. Duplicate determinations made on the same steel, in one case as rapidly as the manipulation could be carried through comfortably, and in the other case, allowing the precipitated oxide to stand in the nitric acid solution 12 hours before filtration, give exactly the same results in both cases; diluting the acid with water after the flask is removed from the fire and then allowing to stand, is disastrous. A direct test showed that in 12 hours standing under these conditions about half the manganese had dissolved. Of course, during standing, the flask should not be exposed to direct sunlight, for fear of formation of nitrous acid.

It does not seem to be necessary to wash the precipitate on the filter with either concentrated or dilute nitric acid, as was formerly directed. As soon as the liquid from the flask has all run through, water may be used at once, for washing both flask, filter tube and filter.

The necessity for the complete removal of every trace of nitric acid by the washing is very great. A very small amount of nitrous or nitric acid in the ferrous sulphate solution, may so interfere with the permanganate titration that the results will be worthless. Large quantities of water may be safely used in the washing and the last drops from the filter tube should be tested with phenolphthalein, or some other delicate indicator.

Whether it is advisable to use the pump or not, during the filtration, seems to be largely a question of the asbestos used. With certain kinds of asbestos a plug ½ in. or more in thickness may be used in the filter tube, and the suction from the pump applied vigorously. With other kinds of asbestos, this procedure results in packing the asbestos so tightly that filtration is retarded, and the subsequent solution of the manganese oxide in the ferrous sulphate solution is also seriously retarded. A mixture of poor asbestos with glass wool works nicely with the pump. Where one operator has six or eight determinations to filter at one time there is no advantage in the pump. With a properly made asbestos filter the liquid will run through as rapidly as it can be poured into the tubes.

The asbestos for filters can be used over and over again, and does not seem to deteriorate from use. After the titration is finished the flasks are emptied into a large beaker, the liquid drained off, and the asbestos washed three or four times by decantation, when it is ready for further use.

For steels containing 0.90 per cent. or over of manganese, it will be necessary to use more than 50 c. c. of the ferrous sulphate solution. In case the manganese oxide does not completely disappear after a little agitation when 50 c. c. of the ferrous sulphate solution has been added, leaving the asbestos perfectly white, add 50 c. c. more of the same solution and agitate again. Of course, the calculations must be varied in accordance with the change.

Very few steels contain enough silicon to cause trouble in the filtration, provided the pump is not used, and it is very rarely necessary to separate silicon before determining the manganese. When the amount of manganese is very large, the filtration is sometimes slow, but if care is taken to keep the tube full of liquid and not to pack the asbestos too tight, there is usually no serious difficulty.

There are some indications that the separated oxide of manganese may not be exactly MnO_2. Comparative experiments on the same steel by the method given above, and by the acetate-bromine method, show thet for steels containing not more than 1 per cent. of manganese, the error, if any, can safely be ignored.

When everything works well, a determination can be made by the method above described, in from an hour, to an hour and a half.

The Determination of Manganese in the Presence of Chromium and Tungsten

Two grams of drillings are dissolved in 20 c. c. of sulphuric acid, (1.2 Sp. Gr.) enough water being added from time to time, to keep the iron sulphate in solution. The solution when completed is oxidized by adding 5 c. c. of nitric acid (1.42 Sp. Gr.) and then evaporated until fumes of sulphuric acid are given off and the carbonaceous matter is destroyed; 100 c. c. of water are added and the solution of the ferric sulphate effected by boiling. The liquid and residue are washed into a graduated 500 c. c. flask and a solution of sodium carbonate added until the solution becomes dark in color and the precipitate formed by the addition of the carbonate dissolves with difficulty.

An emulsion of zinc oxide and water is then run into the flask a little at a time and the flask shaken after each addition until all the iron and chromium have been precipitated. The volume is then made up to 500 c. c. with water and the precipitate allowed to settle; 250 c. c., equivalent to one gram of the sample, of the supernatant liquid is decanted through dry filter and transferred to an Erlenmeyer flask. The solution is acidified with 25 c. c. of nitric acid (1.42 Sp. Gr.) and one gram of sodium bismuthate added. From this point proceed as in the "Bismuthate Method" see "Lord and Demorest" page 84 or in any standard book on steel analysis. Also these notes Method 97.

Nickel—(The Cyanide Method)

NICKEL SOLUTIONS.

1. Potassium Cyanide: 4.4868 grams per liter, plus 5 grams Potassium hydroxide.
2. Silver Nitrate: 2.9250 grams per 500 c. c. water.
3. Potassium Iodide: A 20% solution.
4. Binormal Ammonia: 1 part ammonia to 4 parts of water.
5. Ammonium Chloride: 3750 grams NH_4Cl added to 13.5 liters of water, making about 16 liters. Use 325 c. c. of this solution.

Dissolve 1 gram sample in a 150 c. c. beaker, in 25 c. c. (1.1) HCl and then add 2 c. c. concentrated HNO_3 (or 5 c. c. 1:20 Sp. Gr.) to oxidize all of the iron to the ferric state. Transfer the contents of the beaker to a 500 c. c. graduated flask—containing 75 grams NH_4Cl (ammonium chloride) in 325 c. c. volume. To the flask (1:4) ammonia is added very slowly, until a very pretty red color develops. 50 c. c. concentrated ammonia is now added, the flask and its contents coolen, and diluted up to the mark, and shaken well.

Pour contents of the flask at once on to filters previously prepared and collect the filtrate in a dry, clean, 250 c. c. graduated flask. Fill to the mark.

Transfer the contents of the 250 c. c. flask into a 600 c. c. beaker, add 25 c. c. concentrated HCl which does not quite neutralize all the ammonia. Make the solution slightly acid with sulphuric, and then add ammonia, 1 c. c. at a time until alkaline. Add 2 c. c. of a 20% solution of KI and enough standard silver nitrate from a burette to produce a distinct cloud of silver iodide. Now run in rapidly a standard solution of KCN from a burette, until the cloud of silver iodide disappears, then titrate back with silver nitrate solution until a cloud just forms. One drop will produce this cloud when the end point is reached. Usually about 16 c. c. of standard KCN solution is required for a 3.50% nickel steel.

Standardization of Solutions: KCN and $AgNO_3$.

Dissolve 4.4868 grams KCN and 5 grams KOH in water and dilute to 1000 c. c. The KOH makes the KCN keep better. Also dissolve 2.925 grams $AgNO_3$ in water and dilute to 500 c. c. One c. c. of each will be equal to about .001 grams nickel. To standardize them, weigh out 1 gram of nickel free steel and add 0.6740 grams $NiSO_4\ (NH_4)_2SO_4 6H_2O$, which contains 14.85% nickel and treat just as a nickel steel is treated, up to the point of titration. Make the titration carefully until the turbidity just disappears. Now add 10 c. c. cyanide in excess, and titrate with $AgNO_3$ until a turbidity just appears. This second titration gives the relation between the cyanide and the silver nitrate. For example: Suppose that in the first place, 0.5 c. c. of silver nitrate solution were added and 50.5 c. c. of cyanide solution. Then suppose it took 10.8 $AgNO_3$ to produce a turbidity after the 10 c. c. excess KCN had been added. The cyanide to titrate the nickel would bbe 50.5—(0.5×10/10.8) which is equivalent to 50.04 c. c. Since the amount of nickel present in the portion titrated was 0.3370×.1485 or .050 grams, the strength of the cyanide solution is .050÷50.04 which is equivalent to .0009992 grams nickel per cubic centimeter.

NOTES.

1. The presence of sulphates is necessary to obtain a sharp end point— hence the use of sulphuric acid to neutralize the last part of the large excess of ammonia.

2. Silver iodide is soluble in large excesses in NH_4OH, so care should be taken to have only a small amount of ammonia in excess.

3. Copper and cobalt are the only interfering substances and these are present in steels, usually, in very small amounts.

Dimethylglyoxime Method for the Determination of Nickel in Steel

SOLUTIONS.

1. Hydrochloric acid dissolving solution.
   1 liter concentrated hydrochloric acid (Sp. Gr. 1.19).
   1 liter distilled water.
2. Tartaric acid solution.
   150 grams tartaric acid.
   1 liter distilled water.
3. Dimethyglyoxime solution.
   10 grams dimethyglyoxime.
   1 liter 95% alcohol (ethyl or methyl).

Process of Analysis: Place 1 gram of the steel for analysis in a 150 c. c. beaker. Add 20 c. c. of the hydrochloric acid dissolving solution. Cover the beaker with a watch glass and place it on the hot plate until the sample is all dissolved. Remove it from the hot plate, wash the watch glass and the sides of the beaker with a little distilled water and add 2 to 3 c. c. of concentrated nitric acid (Sp. Gr. 1.42). Boil the solution a few minutes to oxidize the iron and then filter it into a 400 c. c. beaker washing the filter paper five or six times with water. Add to the filtrate plus the washings 40 c. c. of the tartaric acid solution. Make the solution alkaline with ammonia and then slightly acid with hydrochloric acid. Dilute it to about 300 c. c. and heat nearly to boiling. Add 20 c. c. of the dimethylglyoxime solution (for 3.5% nickel steel) and then add ammonia drop by drop with vigorous stirring until the solution reacts slightly alkaline, to litmus. After standing for one hour, filter through a weighed Gooch crucible. Wash the contents of the crucible five or six times with hot water and dry in the oven at 115° to 120° C. for 45 minutes. Cool the crucible in the desiccator and weigh.

The weight of the precipitate multiplied by 20.31 equals the per cent nickel.

Determination of Phosphorus in Steel (Alkalimetric Method)

PROCESS.

Weigh 2 grams sample into a 250 c. c. Erlenmeyer flask, add 75 c. c nitric acid (1.13 Sp. Gr.) boil to complete solution. Add 10 c. c. oxidizing solution potassium permanganate and boil until the pink color disappears and manganese dioxide precipitates. Clear with as small an amount as possible of ferrous sulphate, add 5 c. c. ammonia (Sp. Gr. 0.90) and swirl to dissolve and precipitate formed. Bring to temperature of 80° C., add 50 c. c. ammonium molybdate solution, and shake five minutes.

On cast iron and other irons very high in phosphorus, use smaller samples.

Filter through Shimer filter, washing out the flask with HNO_3 wash, and then with KNO_3 wash, being careful to remove all traces of acid from the flask. Then wash the precipitate with HNO_3 wash several times and 8 or 10 times with KNO_3 wash. Push the felt pad from carbon filter tube, remove the asbestos layer carrying the yellow precipitate and place into the original flask. Care should be taken that no acid comes in contact with the asbestos pad while it is being transferred. Run in about 50 c. c. H_2O free from CO_2, swirl to disintegrate, run in a measured amount of standard NaOH solution. Swirl to dissolve yellow precipitate, add a few drops of phenolphthalein indicator and titrate with standardized HNO_3 to water-white end point. The difference between the number of c. c. NaOH used and the number c. c. HNO_3. used, divided by 100 will give the per cent. phosphorus when a 2-gram sample is used.

SOLUTIONS.

Potassium permanganate for oxidizing use 12.5 grams potassium permanganate to 1 liter of water.

Ammonium Molybdate Solution.

A { 1000 grams Molybdic Oxide.
4000 c. c. Water.
1000 c. c. Ammonia (Sp. Gr. 0.90).

B { 4000 c. c. Nitric Acid (Sp. Gr. 1.42).
6000 c. c. Water.

Mix by pouring A into B a little at a time and agitate constantly. When cool add solution of 0.5 gram microcosmic salt in 25 c. c. water. Let precipitate settle over night and filter through asbestos pad with suction. Solution as used, should be clear.

Standard sodium hydroxide and standard nitric acid solutions.

Dissolve 15.4 grams NaOH as free as possible from Na_2CO_3 in about 2000 c. c. H_2O. Now add a saturated solution of $Ba(OH)_2$ as long as a precipitate forms. Stopper tightly and allow to stand over night. Add a few drops more Ba $(OH)_2$ solution and if no more precipitate forms, filter. If a precipitate does form, allow to stand some time before filtering.

This solution will be a little too strong.

Now prepare a HNO_3 solution by diluting 20 c. c. concentrated HNO_3 to 2000 c. c. Fill a burette with this acid and titrate this against 25 c. c. of the NaOH solution. Now pipette 100 c. c. of Ag_3PO_4 standard solution into a flask, boil down to about 75 c. c. Add 15 c. c. concentrated NH_4OH. Bring to 176° F. (80° C.), add 50 c. c. ammonium molybdate solution and shake five minutes. Filter through a Shimer filter as described above. Proceed to dissolve and titrate as described for steel samples. The difference between the number c. c. HNO_3 required for the NaOH and the number c. c. HNO_3 required to titrate the standard is the number c. c. HNO_3 equivalent to 0.002 grams P. Now add sufficient H_2O to the HNO_3 solution to make 10 c. c. exactly equal to 0.002 grams P. Dilute the NaOH solution until it is exactly equal to the HNO_3 solution. Check former results by running duplicates on the Ag_3PO_4, and also a standard steel of known P. content.

Phenolphthalein Solution. Dissolve 0.2 gram indicator in 200 c. c. 95 per cent. neutral alcohol.

Nitric Acid Wash. Use 1 per cent. solution made by adding 13 c. c. HNO_3 (Sp. Gr. 1.42) to 1 liter of water.

Potassium Nitrate Wash. Dissolve 1 gram KNO_3 in 1000 c. c. H_2O.

Standard Ag_3PO_4 Solution. Dissolve 0.5394 gram pure Ag_3PO_4 in 2000 c. c. 1.13 HNO_3 at 80° F.

The reaction may be written as follows: $12\ MoO_3(NH_4)_3HPO_4 \times 23\ NaOH = 11\ Na_2MoO_4 + (NH_4)_2MoO_4 + Na(NH_4)HPO_4 + 11\ H_2O$.

The best indicator is a dilute alcoholic solution of phenolphthalein and since CO_2 interferes with the end point with this indicator, the NaOH must be free from CO_2, the HNO_3 must be free from CO_2 and also all water used in the titration. Distilled water should be boiled vigorously for some time to expel CO_2 if not certain that the water as it comes from the still is free from it.

If a number of determinations are being run at the same time it is better to filter them all and replace the percipitates in their respective flasks. Dilute and add 25 c. c. NaOH to each. As they are titrated, wash down the inside of the flask with a little CO_2 free water, add indicator and titrate.

The solutions should be titrated against each other, at least once a day and also standardized frequently against the Ag_3 PO_4 solution or a standard steel or both.

The nitric acid, if preserved in a tightly stoppered bottle, will keep its standard indefinitely. The NaOH is subject to slight change, which may be largely prevented by paraffining the inside of the bottle and by using a soda lime guard tube. For greater efficiency and protection, it is probably best, to use soda lime guard tubes on both the NaOH and HNO_3 and siphon the solutions into self-filling zero-overflow burettes, or a pipette for the soda.

When the yellow precipitate is dissolved in the NaOH, the liquid should be kept cool and the solution diluted, as ammonia is set free in the reaction and is liable to be lost by volatilization if the liquid is concentrated. This would cause an error in the HNO_3 titration.

In using the Shimer filter, care should be taken that the plate and felt pads fit evenly in the filter tube, so that when they are pushed from the tube the yellow precipitate will be completely removed. If by chance any of the precipitate should adhere to the filter tube, it should be removed with a piece of moistened filter paper and placed in the flask with the asbestos pad. Sometimes the precipitate creeps badly while washing, and has a tendency to creep over the edges of the tube. This should be removed as above, with a piece of moistened filter paper. Care must be taken not to touch the asbestos pad with the fingers, unless they are free from acid, as any acid on the asbestos would cause an error of titration. It is perhaps best to remove the asbestos with a small spatula and tweezers. The amount of NaOH solution required depends upon the amount of phosphorus present, 10 or 15 c. c. usually being sufficient for low phosphorus but 25 c. c. may well be used for high phosphorus content.

Comparison Method for Phosphorus

Weigh 1 gram sample into a 250 c. c. Erlenmeyer flask, add 75 c. c. nitric acid (1.13 Sp. Gr.) boil to complete solution and expulsion of brown fumes. Add 10 c. c. of oxidizing solution of $KMnO_4$ (12.5 grams to a liter) and boil until pink color disappears and manganese dioxide precipitates. Destroy the manganese dioxide with the least possible amount of $FeSO_4$ (saturate solution usually requires 2 to 3 c. c.) Cool somewhat, and add 5 c. c. of ammonia (Sp. Gr. 0.90) and swirl to dissolve the precipitate formed. Bring to a temperature of 70 degrees Centigrade and add 75 c. c. of ammonium molybdate solution.[4] Shake vigorously for five minutes. The amount of phosphorus in this sample is then estimated by comparison with standards of known phosphorus content, which are prepared each day under the above conditions. The solutions for phosphorus are given in the alkalimetric method, and are the same as used in this method.

MOLYBDIC ACID RECOVERY.

All solutions used containing molybdic acid are poured into a five-gallon crock when the operation is complete, to which is added 2½ cupfuls (about 500 grams) of Na_3PO_4. Heat on the hot plate on an asbestos mat for 24 hours, and stir well. The phosphomolybdate should be completely precipitated in this time. Siphon off the supernatant liquid and pour into an acid-proof sink. Wash the yellow precipitate at least three times with water, siphoning off the liquid each time. The precipitate may now be transferred to a large evaporating dish and dried on the steam plate. To make up molybdic acid solution from the yellow precipitate prepared, use one-half pound measured in cupfuls into a two-liter bottle. Add 800 c. c. distilled water and 600 c. c. ammonia. Allow it to cool, stir well to complete solution, and add 100 c. c. magnesia nitrate mixture to precipitate phosphorus held in connection with the yellow precipitate. (160 grams $Mg(NO_3)_2$ to 400 c. c. H_2O.) Filter into a 16-liter bottle by suction and an alundum cone. It is to be stated here that four 2-liter bottles are prepared from the yellow precipitate at the same time, and all four filtered into the 16-liter bottle. Add to the solution in the 16-liter bottle 7,600 c. c. of (1:1) nitric acid. The usual precautions to prevent precipitation of molybdic acid are taken. The microcosmic salt is then added (30 c. c. of saturated solution), and the clear solution is ready for use.

Method of Determining Titanium in Iron and Steel

Dissolve 5 grams of steel in from 30 to 40 c. c. concentrated hydrochloric acid and 5 c. c. concentrated nitric acid. After evolution has ceased, place triangle under watch glass, and take to dryness on steam plate. Take up with about 15 c. c. hydrochloric acid and from 20 to 30 c. c. water, filter, wash with water and save residue (Res. A). Evaporate filtrate until scum begins to form, and add a little hydrochloric acid (Sp. Gr. 1.11) to prevent separation of ferric chloride on cooling. Transfer to separatory funnel, (150 c. c. capacity) washing out beaker with hydrochloric acid (specific gravity 1.11). Add about equal bulk of ether which has been washed with water. This is best done by shaking up in a separatory funnel. Add ether slowly at first to prevent heating; a good plan is to add a small portion of ether, and cool under tap, after which add balance of ether and shake about eight minutes. Let stand from five to ten minutes and run into second separatory funnel; wash down sides of funnel with hydrochloric acid (Sp. Gr. 1.11) and let stand two minutes and run into second funnel; repeat washing; add ether to second separatory funnel and separate and wash as before drawing off solution into beaker in which steel was dissolved. Heat to drive off ether; cool and add 5 to 10 c. c. hydrogen peroxide to oxidize any iron which may have been reduced during the ether separation and add slight excess of ammonia. This will precipitate the titanium and the balance of the iron. (If this precipitate should contain much iron it will be an advantage to filter, redissolve in hydrochloric acid and make a third ether separation.) Filter the precipitate of iron and titanium; wash with water and dry (Res. B), which can be done by placing funnel in neck of flask and standing on steam plate, or by placing in dry oven. When dry, brush out precipitate onto glazed paper, and burn filter paper together with residue A in a large platinum crucible. After ignition is complete, moisten the residue with a few drops of water, add 4 to 5 drops of concentrated sulphuric acid and 2 to 3 c. c. of hydrofluoric acid, and volatilize by standing crucible on an asbestos pad over a Bunsen burner until sulphuric acid fumes are no longer given off. (Do not heat over direct flame.) Mix residue B, which was brushed onto paper, with about 5 grams of sodium carbonate, (best done by grinding together in a small mortar), and transfer to this crucible, cover, and fuse for about one-half hour; extract with hot water, filter and wash with warm water containing about 2 grams of sodium carbonate to 100 c. c. Without the addition of sodium carbonate to the wash water, the filtrate would be more or less turbid. Sodium titanate and ferric oxide remain on the filter, while phosphates go into solution. Wash residue back into beaker with warm dilute hydrochloric acid, and 10 c. c. concentrated sulphuric acid and take down to sulphuric acid fumes; dilute with water and filter (Filtrate B). In case of much insoluble residue, it may be best to burn filter paper and residue, purify with hydrofluoric acid and sulphuric acid, and fuse with a little potassium bisulphate. After burning the filter paper and residue add about 2 grams potassium bisulphate to the crucible, cover, heat cautiously for 15 to 30 minutes, cool, add 5 c. c. concentrated sulphuric acid, and heat with low flame until fusion becomes liquid. On cooling, it will remain liquid. Pour into a beaker containing 150 c. c. of water, filter and add to filtrate B. Filtrate B, together with filtrate from bisulphate fusion, if any, should be transferred to a 1000 c. c. beaker and neutralized with ammonia. This should be added cautiously toward the end, when the solution turns reddish, adding drop by drop until slight permanent precipitate. Redissolve with a few drops of dilute hydrochloric acid and add about 3 c. c. of ammonia bisulphite solution. Stand in warm place from 10 to 15 minutes. Add 10 to 15 grams ammonium acetate, and acetic acid (Sp. Gr. 1.04) equal to about one-sixth the bulk of solution. Boil 15 minutes, let settle and filter. Wash with water containing a little acetic acid, about 10 c. c. of 50 per cent. acid to 100 c. c. of water, igniting and weigh as titanium oxide (TiO_2) containing 60.05 per cent. titanium.

In cases of small amount of titanium, it is best determined colorimetrically. Proceed same as in the gravimetric method up to the point where filtrate B is obtained. Transfer this filtrate into a 50 c. c. comparison tube, Nessler tube, or some modification if desired and add about 3 c. c. hydrogen peroxide. This developes a yellow color, depending on intensity to the amount of titanium present. Should there be much iron in the solution, a few c. c. of phosphoric acid will counteract the color due to ferric salts. Compare this color with another tube containing water, hydrogen peroxide, about 3 c. c. sulphuric acid and a known amount of standard titanium sulphate solution added until the colors correspond. Comparison can best be made by looking into top of tube. Multiply number of c. c. of solution taken by value of 1 c. c. in titanium and divide by number of grams of steel taken, equals per cent. of titanium. This standard solution may be made up by dissolving 1 gram of titanium oxide C. P. in 30 c. c. hydrochloric acid and 10 c. c. sulphuric acid. Take down to sulphuric acid fumes, cool, dilute with water and filter. Dilute to 1000 c. c. and measure off 10 c. c. of solution. Add water and slight excess of ammonia, filter, wash with water made slightly ammoniacal; ignite and weigh as titaniac acid, containing 60.05 per cent.

Wt. × .6005 divided by 10 equals titanium in 1 c. c. solution.

The Bismuthate—Arsenite Method for the Determination of Manganese in Steel

SOLUTIONS

1. Dissolving solution.
   250 c. c. concentrated nitric acid (Sp. Gr. 1.42)
   750 c. c. distilled water.
2. Standard sodium arsenite solution.
   .6733 grams arsenious oxide.
   2 grams anhydrous sodium carbonate.
   Solution made up to 1 liter.

PROCESS OF ANALYSIS.

Place 1 gram of the steel for analysis in a 200 c. c. flask. Add 60 c. c. of the dissolving solution and place on the hot plate until the sample is completely dissolved and all brown nitrous fumes have been evolved. Remove from the hot plate, cool a moment then add about .25 grams of sodium bismuthate and shake. Continue adding sodium bismuthate in .25 gram lots until a permanganate color is observed which persists even after a few minutes boiling. Next add cautiously a few small crystals of potassium nitrite to dissolve any manganese dioxide which may have been formed by the decomposition of permanganic acid. Boil until all brown fumes are eliminated, then remove the flask from the hot plate and cool to tap water temperature. Bring the solution up to its original volume with cold water, add .5 grams of sodium bismuthate and shake. Allow the solution to stand a few moments and shake again, then filter several times with water. Titrate the filtrate with the standard sodium arsenite solution to the disappearance of the pink color or until no further color change takes place upon the addition of a few extra drops of sodium arsenite solution. The final color is a light green.

REMARKS

To prepare the standard sodium arsenite solution first dissolve the sodium carbonate in about 100 c. c. of distilled water, then add the arsenious oxide and heat until it is completely dissolved. Cool the resulting solution and dilute to 1 liter. The solution should be standardized by titration against a standard steel. It may also be standardized by titration against 25 c. c. of a solution made by dissolving .5754 grams of potassium permanganate in a liter of water. 25 c. c. of this solution contains .005 grams of manganese and should require 25 c. c. of the arsenite solution to titrate it.

Cobalt in Steel

Dissolve 1 to 5 grams sample in 30 c.c. HCl (specific gravity 1.1). When dissolved add a few drops of HF, warm, and oxidize the iron with 1 or 2 c.c. of HNO_3. Evaporate to 10 c.c. Cool and transfer with several portions of 5 c.c. each of 1.1 specific gravity HCl to a 150 c.c. separatory funnel provided with a very short stem, keeping volume under 40 c.c. Extract with 50 c.c. ether saturated with HCl and wash with 5 c.c. of ether saturated with HCl, keeping funnel cold with running tap water during extraction. The chlorides of Va, Ni, Mn, Al and Cu are in the water extract, while practically all the iron is in the ether solution. Drive off ether by heating water extract on steam plate. Heat to boiling and precipitate Cu with H_2S. Filter and wash. Make filtrate alkaline with Na_2CO_3, then faintly acid with acetic acid, and then add 5 grams sodium acetate. Dilute to 100 c. c. heat to 70° C. and pass H_2S through solution for one hour. Filter through a filter paper, on which has been packed macerated filter paper, and wash with a 4% solution saturated with H_2S to remove all manganese sulphide.

Ignite precipitate and paper, dissolve precipitate in concentrated HCl and a little HNO_3, add 5 c. c. H_2SO_4, and evaporate to white fumes.

If cobalt is present in small amounts, (up to 3%), dilute the solution to 100 c. c., add 10 c. c. concentrated HCl and a sufficient amount of nitrose-beta-naphthol solution prepared by dissolving 8 grams in 300 c. c. glacial acetic acid and diluting with 300 c. c. of precipitate to settle several hours, filter, wash with hot 10% HCl, then with water until acid is removed. Ignite, cool, dissolve cobalt oxide in HCl, add H_2SO_4, evaporate to dryness, and ignite at low red heat to $CoSO_4$.

$CoSO_4 \times .38 \div \text{sample} \times 100 = \%\ \text{Co}$.

If the cobalt is present in large amounts, precipitate it with the nickel by neutralizing with NH_4OH and adding 30 c. c. excess of NH_4OH (specific gravity .90), diluting to 100 c. c., and electrolizing on a gauze cathode with a current of 2 amperes. Deposition should be complete in one hour. Weigh nickel plus Co, dissolve in concentrated HCl, and determine nickel by dimethylgloxime method as given in Method 93. Subtract the weight of nickel from the combined nickel and cobalt.

The elestrolytic deposit of Co is generally brown or black and the results tend to be a trifle high. Too small an amount of NH_4OH tends to retard deposition of the metals. Sometimes some of the nickel and cobalt oxides separate out as black specks in the solution. These should be filtered, dissolved, and reprecipitated.

The above method is abstracted from Metallurgical Analysis, by Lord and Demorest.

Oxidation Method for Sulphur in Iron and Steel

As the sulphur is present in these metals in very small percentages only, its accurate determination demands great care. Take from 2 to 5 grams, according to the percentage of sulphur, in a 250 c. c. flask, add 25 to 40 c. c. concentrated nitric acid. If action is too slow, warm to start it. If it is too rapid, place in cold water. The rate of solution must not be too rapid, or low results may follow. A few c. c. of hydrochloric acid may be added from time to time to hasten solution. When all steel or iron is in solution, add 5 c. c. concentrated hydrochloric acid, and evaporate to dryness to render silica insoluble. A temperature of not below 250 degrees Fahernheit should be used, and drying should be continuous until faint odor of hydrochloric acid is perceptible. The usual procedure is to stand on the steam plate over night. Allow to cool, add 20 c. c. concentrated HCl and evaporate until a scum begins to form on the surface of the solution. This operation may be hastened by transferring to a 350 c. c. beaker, as soon as the iron salts are in solution, and evaporating in the beaker instead of the flask. As soon as the scum forms, add from 3 to 5 c. c. of HCl and heat until complete solution. Add 50 c. c. of water and filter through a 9 c. m. filter into a 250 c. c. beaker. Wash two or three times alternately with warm HCl (10%) and cold water. Finish washing with cold water until all iron stains are removed from the filter paper. Add 15 c. c. of a 20 per cent. solution of barium chloride and gently boil for about 15 minutes. Allow this to stand in a warm place for at least three to four hours. Filter through a 7 c. m. filter paper, wash with hot 10% HCl and cold water until all barium chloride is removed. Usually 8 to 10 washings will be sufficient. Place in a platinum crucible, "smoke off" over a low flame, or at mouth of the muffle. Ignite and weigh.

Weight × .1374 = per cent. S.

Rapid Routine
Determination of Silicon in Steel

Weigh 3 grams sample into large casserole and add 6 c. c. H_2SO_4 (Sp. Gr. 1.84) and then 45 c. c. of dissolving mixture. Cover with watch glass and place on hot plate until in solution. It may then be heated over an open flame until it assumes a syrupy condition, and taken to dryness on a hot plate. It should not be heated to a high temperature over an open flame on account of the formation of difficulty soluble oxides. Remove from heat and cool a little, then add about 150 c. c. 10 per cent. solution HCl and heat to solution of all soluble salts. Filter through an ashless paper, using a platinum cone and suction. Wash with hot 10 per cent. HCl and water until free from iron, washing lastly with water. Ignite, cool and weigh as SiO_2. Wt. of $SiO_2 \times .47 \div 3 \times 100$; or wt. of SiO_2 in 3 gm. sample $\times 15.66$ will give the per cent. of silicon.

DISSOLVING MIXTURE.

1000 c. c. Water.
1000 c. c. HNO_3 (Sp. Gr. 1.42).
250 c. c. HCl (concentrated) (Sp. Gr. 1.19).

Method of Determining Silicon in Steel and Cast Iron

APPARATUS AND REAGENTS.

It will be observed that a porcelain beaker and a special oven are designated as the characteristic pieces of apparatus for this method. The oven consists of a copper air bath or oven having a double bottom and lined on the inside with asbestos. The top is heavy copper having six holes to allow the beakers to enter and remain supported by the rim of the beaker. The under side of the top of the oven is also insulated by a layer of asbestos. Provisions are made for holding thermometer to indicate the temperature of the oven.

Glass should not be used for this method, as the high temperature in connection with the acids used causes a disintegration of the glass and leads to unreliable results.

A series of experiments made in porcelain beakers by evaporating the acid mixture and heating to the required temperature for dehydration of the silica, showed no appreciable amount of silica removed from the porcelain by this treatment.

The mixed acids for solution are made by adding 12-½ c. c. concentrated hydrochloric acid, C. P., and 2 c. c. concentrated sulphuric acid, C. P.

Where a number of determinations are to be started at the same time, it is more convenient to mix the quantity of acid required at once and use the proper quantity for each determination. Care should be taken that each one should have the amount of sulphuric acid mentioned, as this is one of the essential features of this method. The dilute hydrochloric acid used for washing is made by adding one part of concentrated acid, C. P., to nine parts of distilled water. Hydrofluoric acid of good quality, particularly free from residue, can now be obtained in the market in ceresin bottles.

OPERATION.

Put one gram of borings in a lipped porcelain beaker holding about 200 c. c. and having a height of about 10 c. m. (Arthur H. Thomas Catalog No. 12512). Add a mixture of 25 c. c. nitric acid, 1.20 Sp. Gr., C. P., 2 c. c. concentrated hydrochloric acid, C. P., and 2 c. c. concentrated sulphuric acid, C. P.

Cover with a watch glass and after the sample is dissolved, place a triangle under the watch glass and evaporate nearly to dryness, preferably on a steam plate, then place the beaker in an oven and heat to 450 degrees F. to 500 degrees F. one hour, or until white fumes of sulphuric acid cease coming off.

Remove from the oven and allow to cool. Add distilled water, about 100 c. c., heat on steam plate with frequent stirring until the salts are dissolved, then add 2 c. c. concentrated hydrochloric acid, C. P., and heat a few minutes, then filter at once, using a policeman and a little filter paper to remove any silicon adhering to the beaker.

Wash with water until most of the iron salts are removed and then with a 10 per cent. solution of hydrochloric acid as long as the washings are colored with iron salts. Finally wash with water until the washings no longer react for chlorine, then ignite and weigh. Treat the contents of the crucible with a few drops of dilute sulphuric acid, C. P., and a few c. c. of hydrofluoric acid, evaporate carefully to dryness, ignite and weigh again. The difference between the two weights is silica, from which the amunt of silicon can be determined.

NOTES AND PRECAUTIONS.

It is essential that the directions in regard to heating in the oven be strictly observd. If this is not properly done, the silica may not be entirely separated, or as has often been demonstrated, may be more or less dissolved during the solution of the salts previous to filtering and in washing on the filter.

Care should be taken to remove all of the silica from the beaker when filtering. A portion of a washed filter paper rubbed over the inside of the beaker by means of a policeman is very efficient for this purpose. The piece of filter paper is washed into the filter and burned with it.

The silica obtained may be contaminated more or less and should be treated with hydrofluoric acid and the proper correction made. Great care should be taken in burning the filter to prevent loss of silica, resulting from air currents of any kind which may readily remove the light silica from the crucible.

A blank test should be made of all the material involved in the determination.

The Evolution Method for the Determination of Sulphur in Iron and Steel

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SOLUTIONS.

1. Hydrochloric acid dissolving solution:—
   1 liter concentrated hydrochloric acid (Sp. Gr. 1.19).
   1 liter distilled water.
2. Zinc chloride solution:—
   35 grams Zinc Oxide.
   100 c. c. concentrated hydrochloric acid (Sp. Gr. 1.19).
   1250 c. c. Ammonia water (Sp. Gr. 0.90).
   1000 c. c. distilled water.
3. Standard iodine solution:—
   3.9687 grams iodine.
   6 grams potassium iodide (free from iodate).
   10 c. c. distilled water.
   Dilute to 1 liter.
4. Starch solution:—
   1 gram potato starch.
   200 c. c. distilled water.

APPARATUS.

The apparatus consists of a 300 c. c. Erlenmeyer flask fitted with a thistle tube and a delivery tube, a tall beaker or drinking glass, and ring stand. The stem of the thistle tube is bent so as to form a water seal and extends nearly to the bottom of the flask. The delivery tube is made of a 25 c. c. pipette and is bent just beyond the stopper so as to slant upward at an angle of about 30° from the horizontal. A few inches beyond the bulb it is bent vertically downward. A short piece of glass tubing attached by means of a rubber connection to the end of the delivery tube extends nearly to the bottom of the tall beaker or drinking glass. The flask is mounted on the ring stand so that heat may be applied from a Bunsen burner.

PROCESS OF ANALYSIS.

Place 5 grams of the steel for analysis in the flask. In the tall beaker put 20 c. c. of the zinc chloride solution and about 200 c. c. of water. Add through the thistle tube 100 c. c. of the hydrochloric acid dissolving solution. Unless the evolution of gas is fairly rapid apply gentle heat to the flask adjusting the flame as nearly as possible so that the acid will boil just as the last particles of steel go into soluon. The evolution of gas should not be so rapid that the individual ıbbles cannot be distinguished as they pass through the zinc chloride ılution. After the sample is all dissolved, boil the solution until ıe end of the delivery tube away from the flask becomes hot to ıe touch, then break the rubber connection, leaving the short glass ıbe in the beaker to be used as a stirring rod. Add to the contents the beaker about 5 c. c. of the starch solution and 25 c. c. of conıntrated hydrochloric acid (Sp. Gr.) 1. 19). Titrate immediately ith the standard iodine solution to a clear blue (not lavender) ılor.

Each cubic centimeter of iodine solution represents .01% sulphur the sample.

REMARKS.

To prepare the zinc chloride solution, first dissolve the zinc ıxide in the concentrated hydrochloric acid, then add 250 c. c. of nmonia water. If the solution is cloudy, filter it. Next add 1000 c. of ammonia water and lastly 1000 c. c. distilled water.

To prepare the iodine solution, dissolve the potassium iodide 10 c. c. of water and then add the iodine. Let the mixture stand ıd shake it from time to time until the iodine is completely disılved. Filter the solution, dilute it to one liter and let it stand for few days before standardization.

To standardize the solution titrate it against the sulphur from standard steel.

To check the titration against a standard steel or in the absence such, the following method may be used: Prepare a solution of ıdium thiosulphate by dissolving 8 grams in water and diluting to ıe liter. Call this solution A. Prepare another solution by disılving .1531 gram of pure dry potassium dichromate in water and iluting to 100 c. c. Call this solution B. Dilute 10 c. c. of solution with about 100 c. c. of water, then add 1 c. c. of the starch soluon and titrate with the standard iodine solution to a blue color hich remains after a few minutes standing. This gives the relation etween the thiosulphate solution A and the iodine solution. Next ilute 10 c. c. of solution B with 50 c. c. of water, then add .5 gram potassium iodide and when it is all dissolved, add 5 c. c. of conıntrated hydrochloric acid. Let this solution stand for about five ıinutes, then dilute to 100 c. c., add 10 c. c. of solution A and 1 c. c. starch. If a blue color is observed, add a few more c. c. of soluon A, then titrate immediately with the standard iodine solution the appearance of a blue color.

To calculate the sulphur value of the iodine solution, divide the c. of iodine used in the first titration by the c. c. of thiosulphate used in the first titration. Multiply the result by the c. c. of thiosulphate used in the second titration and subtract the c. c. of iodine solution used in the second titration. The result divided into .005 equals the number of grams of sulphur equivalent to 1 c. c. of the iodine solution.

The starch solution is made by boiling 1 gram of potato starch with 200 c. c. of water for a few minutes. It should be made fresh every few days.

Tungsten, Silicon and Chromium in High Speed Steel

PREPARATION OF SAMPLE

Anneal sample 2 hours at 1400° F. Cool slowly and reject outside surface.

TUNGSTEN AND SILICON

Dissolve 2 grams in a 200 c. c. casserole in 40 c. c. HCl (specific gravity 1.19) by heating nearly to boiling. Add a few drops HNO_3 drop by drop, at intervals until sample as decomposed. Add 5 c. c. HNO_3, boil down to 10 c. c., add 50 c. c. water and boil several minutes and filter. Wash well with 5% hot HCl until $FeCl_3$ is removed. Evaporate filtrate to dryness, take up in 15 c. c. HCl (specific gravity 1.19) until iron salts are dissolved, dilute to 50 c. c., boil, filter, and wash out iron. Ignite the two precipitates in a platinum crucible, being careful not to heat above a red heat. Cool and weigh.

Add 3 drops of sulphuric acid and 5 c. c. HF and evaporate acids and ignite at low red heat. Loss equals SiO_2.

$SiO_2 \times .4693 \div \text{sample} \times 100 = \%\ Si$.

The residue is impure WO_3. To purify it, fuse it with 5 grams Na_2CO_3, dissolve and filter off residue. Wash with hot water, ignite, and subtract from weight of impure WO_3.

$WO_3 \times .793 \div \text{sample} \times 100 = \%\ W$.

CHROMIUM

Add 15 c. c. H_2SO_4 plus 15 c. c. HNO_3 to filtrate and evaporate to white fumes. Add 25 c. c. HNO_3, dilute to 200 c. c., boil until salts are dissolved. Oxidize with strong $KMnO_4$ until permanent color remains after boiling. Decompose excess permanganate by boliing 20 minutes. A precipitate of MnO_2 should remain. If it does not, add more $KMnO_4$. Keep volume of solution approximately constant. Filter off MnO_2, cool to room temperature, dilute to 350 c. c., add an excess of standard ferrous sulphate and titrate excess with standard $KMnO_4$ to a pink color which remains 1 minute. The ferrous sulphate used minus the permanganate used multiplied by .001733 (if $N/_{10}$ solutions are used), equals weight of Cr present. $Cr \times 11 \div \text{sample} = \%\ Cr$. If $N/_{10}$ solutions are not available, the Fe value of the solutions multiplied by .31 equals the Cr. value.

Cast Iron

CARBON IN CAST IRON

Total Carbon—One gram sample. Run same as carbon in steel.

Free Carbon—Dissolve 1 gram sample in 50 c. c. 1 to 1 by volume HCl and boil 20 minutes. Filter on prepared and blasted gooch, dry in oven and run pad plus precipitate same as carbon in steel.

Combined carbon is equivalent to total carbon—free carbon.

PHOSPHORUS IN CAST IRON

One gram sample. Dissolve same as in steel. Filter. Run same as phosphorus in steel.

SULPHUR AND MANGANESE

Run same as in steel.

NOTE: In some cases some chemists prefer to use the Oxidation Method for Sulphur. But if the specification under which it is purchased, is based on the Volumetric Method, it should be used, as the methods give different results.

Thermometers

$$\text{Fahrenheit}^\circ = \frac{\text{Centigrade}^\circ \text{x} 9}{5} + 32. \qquad \text{Centigrade}^\circ = \frac{\text{Fahrenheit}^\circ \text{-} 32 \text{x} 5}{9}$$

Ready Reference: Comparison of Centigrade and Fahrenheit Thermometer Scales

C°	F°	C°	F°	C°	F°	C°	F°	C°	F°	C°	F°
—40	—40	7	44.6	38	100.4	69	156.2	99	210.2	245	473
—35	—31	8	46.4	39	102.2	70	158.0	100	212.0	250	482
—30	—22	9	48.2	40	104.0	71	159.8	105	221.0	255	491
—25	—13	10	50.0	41	105.8	72	161.6	110	230.0	260	500
—20	— 4.0	11	51.8	42	107.6	73	163.4	115	239.0	265	509
—19	— 2.2	12	53.6	43	109.4	74	165.2	120	248.0	270	518
—18	— 0.4	13	55.4	44	111.2	75	167.0	125	257.0	275	527
—17	1.4	14	57.2	45	113.0	76	168.8	130	266.0	280	536
—16	3.2	15	59.0	46	114.8	77	170.6	135	275.0	285	545
—15	5.0	16	60.8	47	116.6	78	172.4	140	284.0	290	554
—14	6.8	17	62.6	48	118.4	79	174.2	145	293.0	295	563
—13	8.6	18	64.4	49	120.2	80	176.0	150	302.0	300	572
—12	10.4	19	66.2	50	122.0	81	177.8	155	311.0	310	590
—11	12.2	20	68.0	51	123.8	82	179.6	160	320.	320	608
—10	14.0	21	69.8	52	125.6	83	181.4	165	329.	330	626
— 9	15.8	22	71.6	53	127.4	84	183.2	170	338.	340	644
— 8	17.6	23	73.4	54	129.2	85	185.0	175	347.	350	662
— 7	19.4	24	75.2	55	131.0	86	186.8	180	356.	360	680
— 6	21.2	25	77.0	56	132.8	87	188.6	185	365.	370	698
— 5	23.0	26	78.8	57	134.6	88	190.4	190	374.	380	716
— 4	24.8	27	80.6	58	136.4	89	192.2	195	383.	390	734
— 3	26.6	28	82.4	59	138.2	90	194.0	200	392.	400	752
— 2	28.4	29	84.2	60	140.0	91	195.8	205	401.	420	788
— 1	30.2	30	86.0	61	141.8	92	197.6	210	410.	400	824
0	32.0	31	87.8	62	143.6	93	199.4	215	419.	460	860
1	33.8	32	89.6	63	145.4	94	201.2	220	428.	480	896
2	35.6	33	91.4	64	147.2	95	203.0	225	437.	500	932
3	37.4	34	93.2	65	149.0	96	204.8	230	446.	520	968
4	39.2	35	95.0	66	150.8	97	206.6	235	455.	540	1004
5	41.0	36	96.8	67	152.6	98	208.4	240	464.	560	1040
6	42.8	37	98.6	68	154.4						

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